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Chemical engineering

CHEMICAL ENGINEERING OPERATIONS

An Introduction
to the Study of Chemical Plant

BY

FRANK RUMFORD
Ph.D.(GLAS.), B.Sc.(LOND.), M.I.Chem.E., F.R.I.C.



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PREFACE

THE purpose of this book is to provide an introduction to those processes which are peculiar to chemical plant practice. For this reason, no specific section is devoted to fluid flow, while power generation and transmission, with the kindred process of refrigeration, are not discussed. The general method of treatment is to survey the plants which have been developed to carry out the process under review, and to give a short analysis of the underlying principles without any attempt at exhaustive treatment. Examples are then given, if necessary, to illustrate the design of plant from these principles, and it has been the author's endeavour to give some practical basis for design from first principles in all cases. Long citations from published working figures have deliberately been avoided.

The book is designed as a textbook for student use, and not as a comprehensive treatise. Full treatment of any one of the processes described herein demands a separate volume of at least the bulk of the present work. Nevertheless, it is hoped that the simple treatment outlined here may be of considerable assistance to chemical technologists who are seeking a clear mental picture of the basic principles underlying chemical plant operation.

The author wishes to acknowledge the able assistance of Dr. G. Edwards, of the Royal Technical College, Glasgow, who has made the drawings from which the illustrations in this book are prepared.

F. R.

Glasgow,
July 1949

CHAPTER I

CHEMICAL WORKS PUMPING

The transportation of all types of fluid by means of pressure differences is at once the simplest and most fundamental of all unit processes operating in chemical plant. It is necessary to consider the movement of material of widely varying physical properties and often of extremely corrosive nature. The pump in a chemical plant is frequently the weakest link in a chain of corrosion resistance, and always demands a major share of the attentions of plant mechanics. If a pumping system is neglected, it is one of the most probable causes of an enforced shut-down, while nothing causes more annoyance than a leaking or inadequate pump service.

There are three main classes of pump used in chemical works :

(1) Reciprocating, or ram pumps, with valves.
(2) Centrifugal pumps, without valves.
(3) Rotary pumps with incorporated slide-valves.

Class (1) is primarily for high pressures and low throughputs. Class (2) is designed for low pressures and high throughputs. Class (3) represents a compromise between 1 and 2.

Outside these main systems are certain special devices, such as jet or entrainment pumps. All find application in both liquid and gas pumping, but the details of construction differ rather widely, and it is proposed to deal first with liquid and then with gas pumping.

Liquid Pumping. Liquids have been pumped in reciprocating pumps for a very long time, the history of water pumping in such units going back for over 2000 years. The essential features of such pumps were settled long ago, and a simple belt-driven, single-acting model of the type shown in Fig. I 1 shows these principles clearly. This pump was designed to force a suspension of solids into a filter press, at pressures of the order of 100 lb./sq. in. The long plunger slides past the fixed packing at a relatively slow speed —50 to 100 ft./min.— and the simple ball-valves are adequate in these conditions to give a volumetric efficiency of 80-90%. This

efficiency is almost independent of discharge pressure and is not greatly affected by variations, within reasonable limits, of the pressure on the suction side. The overall mechanical efficiency of such a pump may be of the order of 50%, with a slurry containing up to 20% of solids. With slight modifications a pump of this kind will force liquids up to a pressure of 5000 lb./sq. in. in a single stage. If the liquid is not corrosive the amount of maintenance required is small and the pump will last almost indefinitely ; wear will be more marked when slurries are pumped, but even then this is shown mainly in a gradual falling off in the volumetric efficiency.

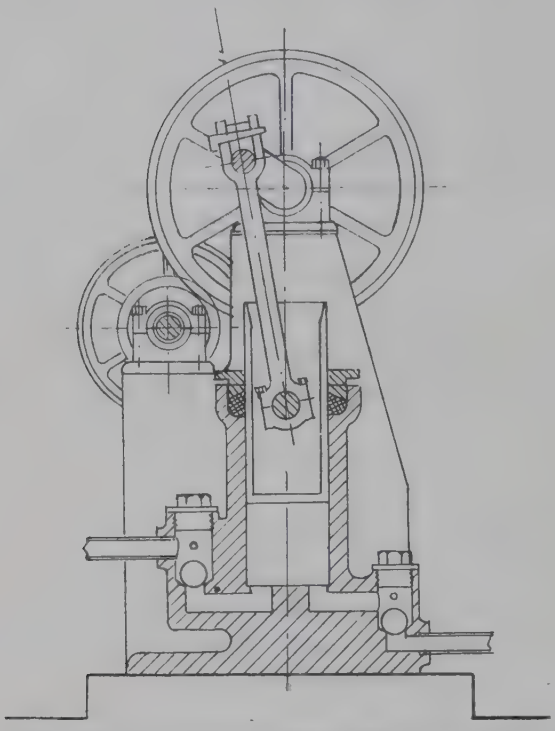


Fig. 1–1.—Reciprocating Plunger Pump.

The pump of Fig. 1–1 is a single-acting, single-cylinder pump, and despite a large air chamber fixed on the outlet line will give a pulsating outlet stream. This will strain the joints on a pipeline, and, in the particular case of a filtration slurry, may impair the working of the filter unit. The discharge can be made more even by using double-acting pumps, which pump at both forward and backward movement of the ram ; in addition the pump can be made up with two cylinders in parallel, so that there are four discharge strokes in each complete cycle. This is the most generally used type of reciprocating pump used today, and is often connected for direct steam drive, as shown in Fig. 1–2. This shows a " piston " pump, with packing on the actual ram, as distinct from the " plunger " type with fixed packing past which the ram moves. As the complexity of the pump increases, the field of application grows more restricted, and the form of Fig. 1–2 would not be suitable for slurries.

The reciprocating pump, in these and several other modifications, gives a volumetric efficiency of 65–95%, and a mechanical efficiency of more than 50%. When working on non-corrosive liquids the

pump is almost everlasting, as wear on the slow-moving parts is slight. Nevertheless, it presents an abundance of points for attack by chemicals. The good fit needed at the glands and valves is soon destroyed by any mildly corrosive agent, and the relatively complicated construction means a high initial cost and requires a good constructional material, which is generally not well adapted to withstand chemical attack. Against this must be set the absolute reliability and positive action which are unique to reciprocating pumps.

Calculation of the output of these pumps is simple. The volume swept out by the ram, with due regard to single or double action, multiplied by the number of strokes per minute, gives the theoretical capacity of the pump. This is never quite attained in practice, due to lag in valve action, slip past the ram, and slight volume changes

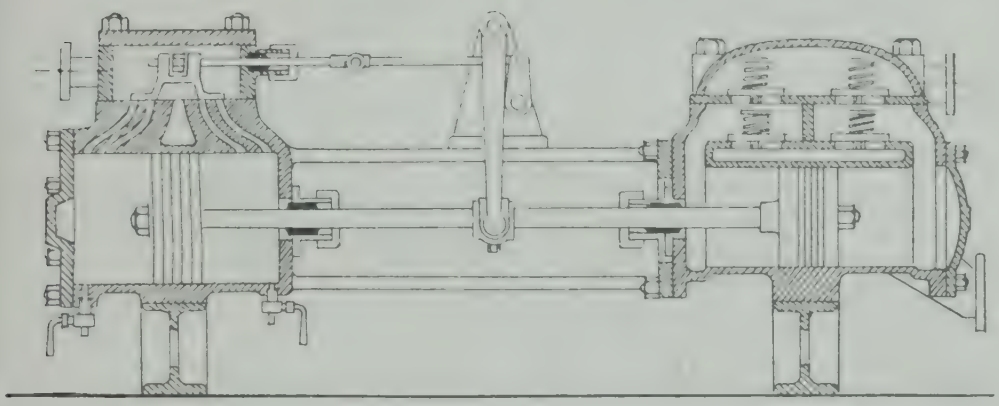


Fig. I–2.—Steam-Driven, Double-Acting Piston Pump.

under pressure. A slow-moving pump of piston speed 50 ft./min. should give a volumetric efficiency of over 90%, while a high-speed pump working on a mobile liquid of high vapour pressure may only work to 50% of the calculated figure. Mechanical efficiencies—the actual work done in foot-pounds compared to the energy expended – will vary more widely, especially with direct-drive steam pumps. It is difficult to give any definite figure, as so much depends on the mechanical losses by friction, but in good practice a figure of 50% can usually be obtained.

The range of liquids handled in chemical plants is far wider than in any other sphere of industrial activity. Liquid pumps must deal with fluids as viscous as glycerine or as mobile as alcohol, as dense as "heavy liquid suspensions" or as light and volatile as aviation spirit. The pressure required to pump these from place to place

will vary enormously, and it is essential to choose, from the wide array of formulae available, those which are derived from basic principles rather than some of the many which have been produced to fit the needs of one particular industry. The fundamental formula for pressure drop in a pipe-line due to fluid friction is given by

$$P = 8\left(\frac{R}{\rho v^2}\right)\frac{L}{d}\cdot\frac{\rho v^2}{2g}$$

where P = pressure drop in lb./sq. ft.
R = frictional force per unit area of wetted surface.
ρ = fluid density in lb./cu. ft.
v = velocity of flow in ft./sec.
L = length of pipe in feet.
d = diam. of pipe in feet.
g = gravitational constant—32·2 ft./sec./sec.

The expression is self-consistent, in that C.G.S. values may be substituted, with $g = 981{\cdot}5$, to give a value for the pressure drop in gm./sq. cm.

The correct value for the composite factor $R/\rho v^2$ must be computed from the value of Reynolds' Criterion—$vd\rho/\eta$ or Re. In this factor, η is the viscosity of the fluid, while v, d, and ρ have the same significance as before. When the value of Re has been calculated from the data available, the value of $R/\rho v^2$ can be read from experimental curves, as in Fig. I–3, connecting these two variables. Such curves clearly fall into two sections, one the common straight line for viscous flow, and the other a series of curved lines relating to various pipe systems with eddying fluid flow. When the flow is viscous, or stream-line, the connection between the two factors is given by

$$\left(\frac{R}{\rho v^2}\right) = \left(\frac{8}{vd\rho/\eta}\right)$$

This region is terminated, for circular pipes, by a value for Re of 2000 ($R/\rho v^2 = 0{\cdot}004$), which corresponds to a very low value of fluid velocity for most industrial purposes. The second region, of eddying or turbulent flow, is not governed by any exact relationship and varies widely with the "roughness" of the pipe being used. Thus for $Re = 1 \times 10^6$ the value of $R/\rho v^2$ may vary from 0·0015 up to 0·007 as the pipe surface alters from smooth brass to a heavily corroded iron. Choice of the appropriate value must thus be made quite arbitrarily, with a knowledge that values may vary widely in the course of time.

This gives a method for the determination of pressure drop due to fluid friction for straight cylindrical pipes. If the pipe is not cylindrical, the value for d in the expressions above can be replaced by the value of $4m$, where m is the hydraulic mean depth. This substitution of the ratio of channel cross-section to wetted perimeter is an approximation, and there is little experimental evidence to show whether change from viscous to eddying flow occurs at the same value for $v4m\rho/\eta$ as for $vd\rho/\eta$. However, the accuracy of calculation is in any case doubtful, and the substitution of $4m$ for d has been generally accepted. For stream-line flow between parallel planes, which may be regarded as the extreme divergence from flow

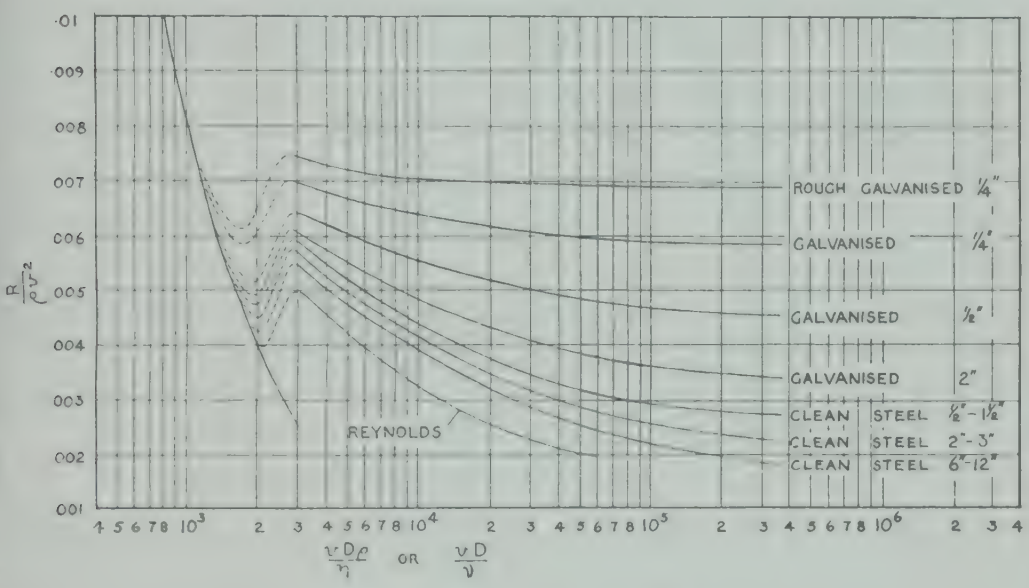


Fig. I–3.—Typical Experimental Curves, $R/\rho v^2$ v. $vd\rho/\eta$.

in a circular pipe, the value of $R/\rho v^2$ is equal to $12\eta/v4m\rho$ and similar changes will probably occur in turbulent flow.

In addition to pressure drop due to friction, the work done by a pump must balance pressure drop due to rise or fall of the fluid between inlet and outlet of the pipe system, changes in velocity head, friction losses due to bends, elbows, valves, and similar obstruction, and possible volume changes of the fluid—the bulk relation generally expressed as Bernoulli's Theorem of Energy Conservation. The increase in height of the liquid above a datum line may, of course, be the main load on the pump, but other pressure drops should be relatively small. They are usually expressed in terms of "velocity heads" ($\rho v^2/2g$) or "pipe diameters". Tables

for typical losses can be found in most reference works on pumping, but a few typical values are set out below :

Right-angle elbow turn	1 velocity head
Right-angle curved bend	0·5 ,, ,,
Globe valve	2 ,, ,,
Tee-piece bend	1 ,, ,,
Entrance loss, pipe from tank	1 ,, ,,

When a gas is being pumped, the same general equation applies when the pressure drop P is negligible compared to the total pressure of the gas. The value of the velocity v and the density of the fluid ρ will remain appreciably constant over the whole length of the pipe-line. The same relation will then exist between the friction factor $R/\rho v^2$ and the value of Reynolds' Criterion, while similar values can be taken for the pressure drop due to bends and fittings.

Example. *Glycerine, of density* 1·26 *gm./ml. and viscosity* 8 *poises, is being pumped from a tank at ground level to a vessel* 20 *ft. above at a rate of* 10,000 *lb./hr. The length of* $1\frac{1}{2}$*-in. pipe used is* 100 *ft. ; and it is bent twice at right angles. Calculate the power requirements of the pump.*

The volume of glycerine pumped per second is 0·0354 *cu. ft. and the pipe cross-section is* 0·0122 *sq. ft. Hence* $v = 2{\cdot}91$ *ft./sec., and the value of* $vd\rho/\eta$ *will be*

$$\frac{2{\cdot}91 \times 0{\cdot}125 \times 78{\cdot}5}{0{\cdot}537} = 53$$

(*Notice that all values are expressed in F.P.S. units.*) *The flow is therefore viscous, and*

$$\frac{R}{\rho v^2} = \frac{8}{53} = 0{\cdot}151$$

Then

$$P = 8 \times 0{\cdot}151 \times \left(\frac{100}{0{\cdot}125}\right) \times \left(\frac{78{\cdot}5 \times 2{\cdot}91^2}{64{\cdot}4}\right)$$

$$= 10{,}000 \text{ lb./sq. ft. } (70 \text{ lb./sq. in.})$$

The additional drop for two bends will be, at most,

$$2 \times \left(\frac{78{\cdot}5 \times 2{\cdot}91^2}{64{\cdot}4}\right) \text{ or } 20{\cdot}7 \text{ lb./sq. ft.}$$

The fluid pressure due to 20 *ft. of liquid of density* 78·5 *lb./cu. ft. will be* 1570 *lb./sq. ft.*

The total pressure, ignoring other small losses, against which fluid is pumped will be 11,590 *lb./sq. ft., and* 127·5 *cu. ft. are pumped per*

hour. This is a rate of 21,600 ft.-lb. min. or 0·75 h.p.; assuming a 50% overall efficiency, a motor of 1·5 h.p. would be installed. Incidentally it might be much cheaper to heat the glycerine before pumping and lower the fluid viscosity.

When an especially corrosive or abrasive mixture is to be pumped by a reciprocating pump, a number of modifications to the standard

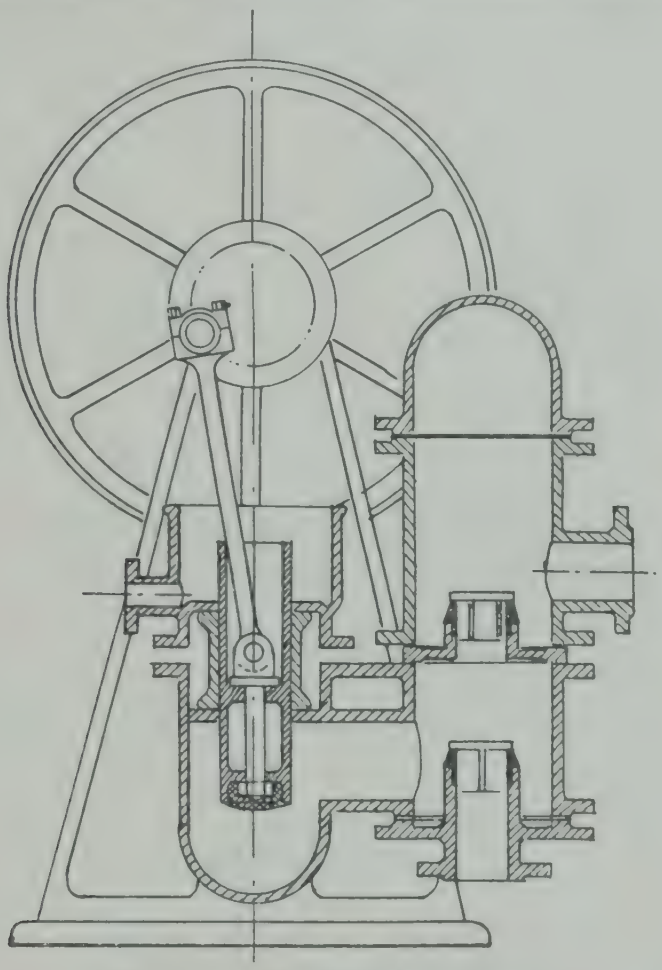


Fig. I–4.—Kestner Acid Pump.
(Courtesy of Kestner Evaporator and Engineering Co. Ltd.)

form have been suggested. Fine machined surfaces must not be relied upon, and very simple valve systems chosen, while the high volumetric efficiency will rapidly fall off unless special arrangements are made to isolate the moving parts from the corrosive fluids. Sulphuric acid, in various strengths, is a typical corrosive chemical which must still be handled in very large quantities, and a number of pumps have been devised for this purpose. The Kestner acid pump shown in Fig. I 4 deliberately allows for a low volumetric

efficiency. The cast-iron or silicon-iron plunger, working inside a cylinder of similar material, is so dimensioned as to give a small leak of acid past the working surfaces. This acid collects in a cup ring at the cylinder top, and drains back to the pump suction. The valves are of a simple cylindrical design, seating by dead weight, and corrosion by the acid, especially if this is over 75% H_2SO_4, is small. Another pump designed primarily for sulphuric acid pumping, but with a very wide range of possible concentration, is the

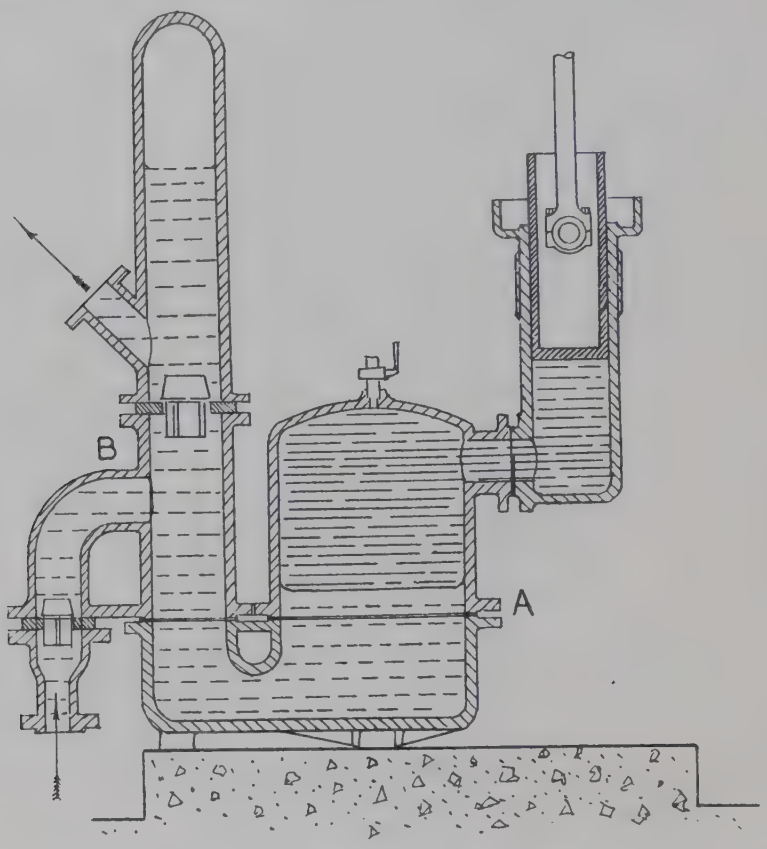


Fig. I–5.—Ferraris Acid Pump.

Ferraris type shown in Fig. I–5. The plunger of this pump works in an oil chamber and the pressure pulsations are transmitted to a balance chamber (A of Fig. I–5) which is partly filled with oil; below the oil is the acid, also following pressure fluctuations, which are conveyed to a lead valve chamber B with hard lead valves. The pump must work at slow speeds (5–10 r.p.m.) or oil–acid emulsions form, so that the Ferraris is large and clumsy for a given throughput of acid. Nevertheless, these pumps are very free from mechanical troubles, and have a long record of even working in the

sulphuric acid industry. A Ferraris pump delivering 1000 gal./hr. of 60% acid to a head of 80 ft. had an overall mechanical efficiency of just on 50%.

When the mixture being pumped would emulsify rapidly with oil, but does not attack rubber or some similar material, the oil "diaphragm" of the Ferraris can be replaced by a flexible disc or

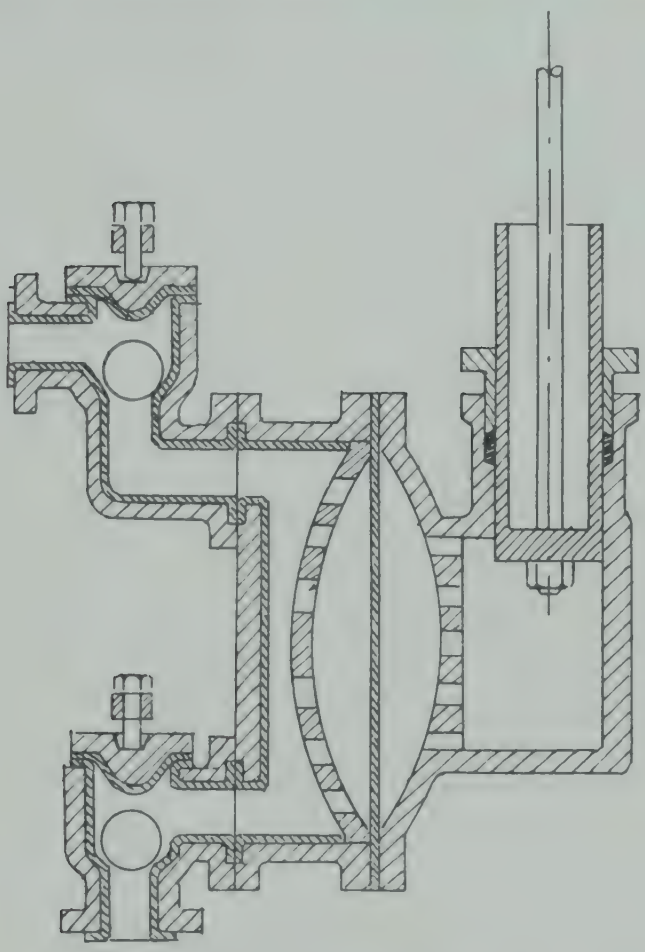


FIG. I-6.—DIAPHRAGM PUMP.
(*Courtesy of Nordac, Ltd.*)

tube, moving generally between guide-plates. The plunger, working in oil or water, is on one side of the diaphragm, and the corrosive solution in a suitable valve system on the other; a pump of this type, designed to handle hot pickling solutions containing appreciable quantities of free HCl, is shown in Fig. I-6. A diaphragm pump of somewhat different design is generally used when pumping slurries with large quantities of abrasive solids in suspension against a small fluid head. A discharge valve of simple design, seating by dead

weight, with no attempt at close fitting, is carried in the centre of a flexible rubber sheet mounted over a cone-shaped suction chamber with a corresponding suction valve at the base. As the discharge valve moves up and down, at about 25 strokes per minute, it will give a suction lift of about 15 ft., sufficient to lift a thick sludge from the base of a thickening tank to a rotary vacuum filter or some similar process plant. The stroke of these pumps is short—about 1 in. on a 12-in. diameter diaphragm—in order to diminish wear on the flexible material. They will run without trouble for long periods

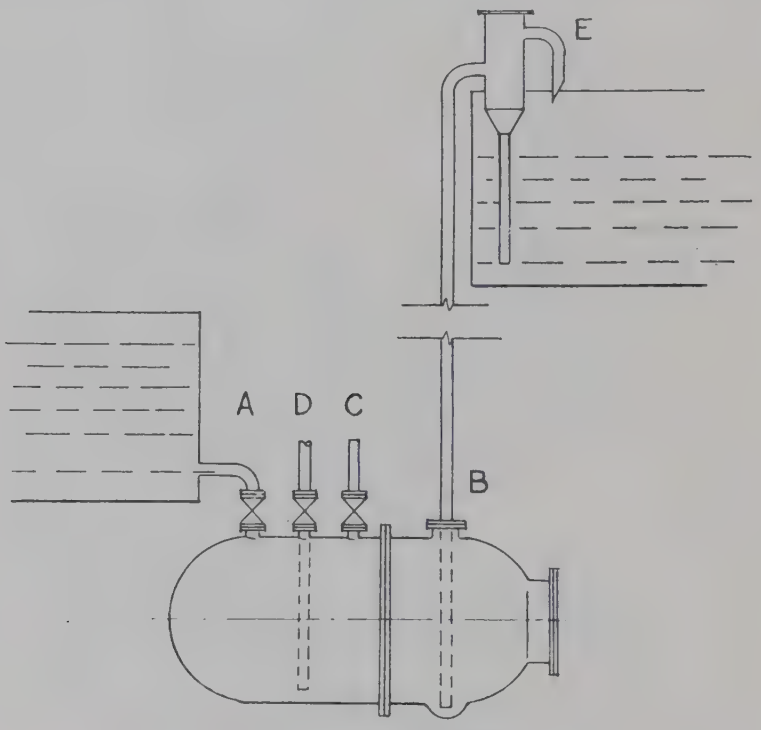


Fig. I–7.—Acid Egg.

of uninterrupted service, but if shut down without draining out all slurry, the valves tend to stick on restarting. Volumetric efficiency is high, as the slurry forms a self-sealing deposit round the valve-seats.

The solid ram, with eccentric drive, common to all the pumps described above, may be replaced by a ram of compressed air. In the simple form of a pressure "egg" this process was once very widely used, and may still be met with, though now considered almost obsolete. The acid egg, as shown in Fig. I–7, was very suitably employed in lifting a charge of acid for a batch nitration. The cast-iron cylinder, in sizes up to 4 ft. in diam. by 10 ft. long,

was filled from storage tanks, or from carboys, with a weighed charge of nitration acid. This could be mixed by opening air-valve D and allowing air to blow freely from the vent C. The mixed acid was then raised by lift pipe B to a tank at the top of the nitration building by closing C and inlet valves A. At the top of the lift, the acid discharged through a splash head E, but as the last of the charge went up the pipe it was followed by a rush of air with some spraying and acid-air losses. The efficiency in any case was low, as a minimum amount of compressed air equal to the egg volume was discharged to waste. Nevertheless, the extreme simplicity of the acid egg will always make it a possible last resort when extremely corrosive solutions must be pumped to a height at intervals. In these circumstances, it is probable that a single hand-operated acid egg will show an efficiency of 10–20% on power fed to the air-compressor.

Several attempts have been made to improve on the simple pressure egg, a very typical instance being the Tungstone unit shown in Fig. I–8. This is really two small eggs placed side by side, and operated in turn by the rotating valve A, which is driven by an independent motor. As this valve rotates at about 10 r.p.m. it opens one vessel to atmosphere, allowing it to fill by gravity from a static feed tank, while the other is simultaneously put under air pressure, forcing the liquid charge up the pipe-line. A pump of this kind can be used for hot oil, using steam under pressure in place of the air, with considerable economy in power.

Positive displacement pumps of any kind possess the cardinal virtue of certainty. If the displacing agent moves through a specified volume, then the fluid which has previously occupied this space must go somewhere, and there are few limits to the conditions which can be put on the outlet stream. As against this, discharge always tends to be intermittent, the first cost and depreciation rate are high for any given throughput as compared to a centrifugal or rotary pump, and it is sometimes difficult to construct a complicated reciprocating pump system in corrosion-resisting material.

The centrifugal pump consists of a rotor, carrying as a rule 6 to 12 blades, spinning at a high speed inside a fixed casing. Liquid is drawn in at the centre, sweeps along the curved blades, and is discharged at the periphery of the rotor; there are no valves, and, at first sight, no close-fitting surfaces to wear rapidly in the presence of corrosive liquids. In Fig. I–9 a typical acid-resisting centrifugal

pump is shown in section ; the rotor is in high-silicon iron, with a stainless steel shaft. It is of the closed impeller type, in which the curved radial blades are enclosed between front and back plates, as shown in Fig. I–10b. Such closed impellers give a better conversion of mechanical energy into fluid head, but are less suitable than the open impellers of Fig. I–10a for pumping slurries. Even with a closed impeller, the design of a single-stage centrifugal pump is

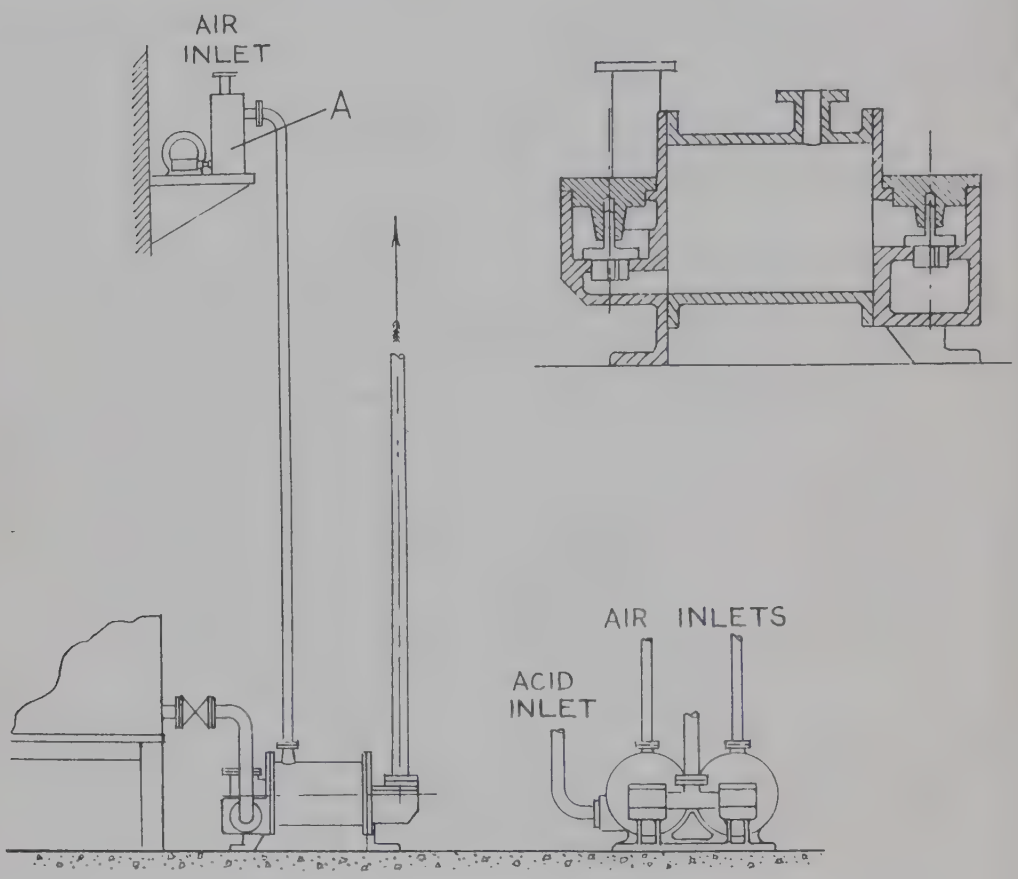


Fig. I–8.—Tungstone Pump.

(*Courtesy of Tungstone Products, Ltd.*)

simple, and it is easy to construct in almost any type of corrosion-resisting material, from stoneware to high-silicon iron. As would be expected, the initial cost is low—about one-fifth of the cost of a reciprocating pump for a similar duty—and the ease with which they can be coupled direct to small electric motors is another factor which has made for a steady increase in the use of these pumps over the last twenty years.

All centrifugal pumps are designed for some specific duty. Ideally, a liquid enters the centre, or eye, of the impeller and flows

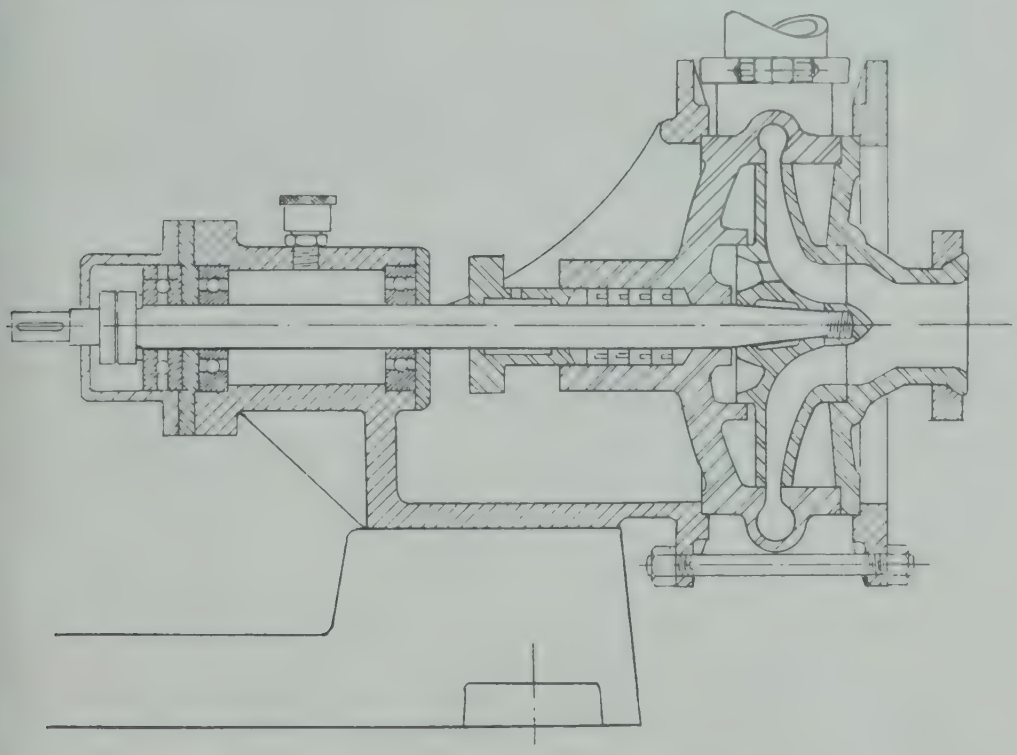
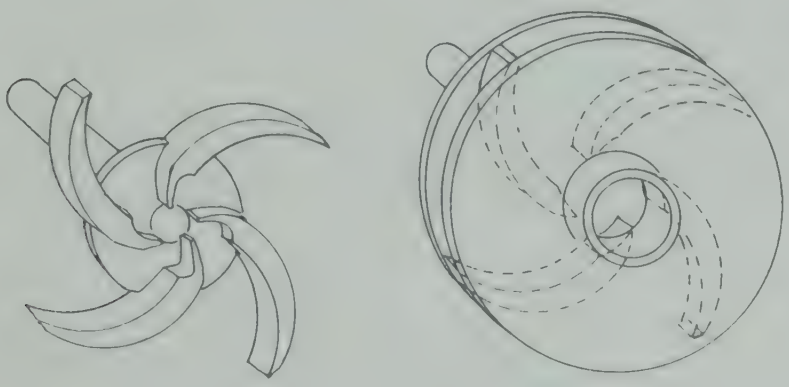


Fig. I–9.—Centrifugal Pump for Acids.

out to the blade tip, leaving at a velocity compounded from blade-tip motion and the movement of the liquid along the blade. This



(a) (b)

Fig. I–10.—Centrifugal Pump Impellers.

velocity head is then to be converted as efficiently as possible into fluid pressure in the discharge line. In order to obtain very

high efficiencies of conversion it is necessary to surround the rotor with a static ring of curved vanes, as in a water turbine. This complicated construction is not a practical proposition with the corrosive liquids to be handled in chemical plant, and a compromise is made between simplicity of design and pumping efficiency. The characteristics of a centrifugal pump are best expressed in a series of curves, as shown in Fig. I–11. When the pump is run at constant speed, and the outlet pressure, or head, gradually increased, then the output, power, and efficiency follow the lines of 11a ; if the pump is discharging at a constant head and the speed is increased, then the output, power, and efficiency follow the diagram 11c ; while constant output with varying speed shows head, power, and efficiency changes as in 11b. For any one pump there is a definite set of conditions for which the pump efficiency is at a maximum, and the centrifugal is therefore not nearly so flexible in operating conditions as the average reciprocating pump. It may be noted as an approximate rule that for any pump, output varies as speed, head as square of speed, and power required varies as the cube of speed.

The apparent simplicity of a centrifugal pump disguises a number of drawbacks. In the first place, a single-stage unit is severely limited with respect to head, or outlet pressure. Small pumps, of diameters below 6 in., will not usually develop more than 25 ft. of fluid head, and it is rare to find any single-stage pump giving more than 100 ft. head. This limitation is inherent in the conversion of practicable fluid velocity to pressure head, and can only be overcome by building the pump in more than one stage. The inlet of a second stage takes the pressure given by the first, and so on, and it is quite possible to construct pumps with ten or more stages. The simple construction so necessary in pumping corrosive chemicals has now disappeared, and in consequence the multi-stage centrifugal is only used in hydraulic work. Again, although the pump has no valve systems, the rotor must be carried by a shaft which revolves at high speed in a close-fitting gland, and this is generally exposed to the action of the liquid being pumped. The wear and tear at this point is often very considerable, and it is almost impossible to keep the gland of a centrifugal pump tight, although a number of special packings have been used. In order to check this leakage, " lantern " rings are incorporated into the packing, with a pipe to feed lubricants or inert diluents into the middle of the gland channel. This puts a

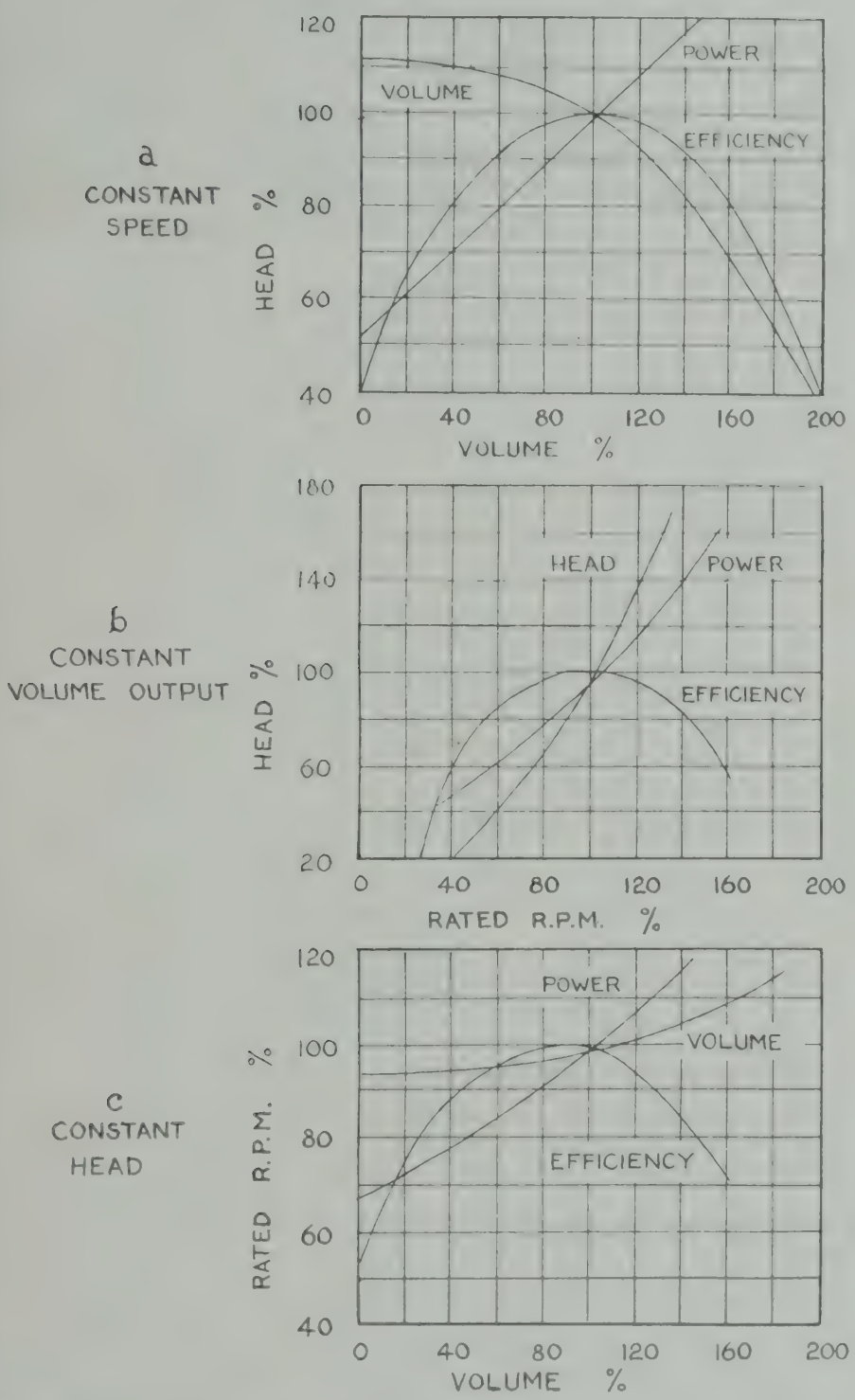


FIG. I–11.—CHARACTERISTIC CURVES FOR CENTRIFUGAL PUMP.

certain amount of the added material into the liquid being pumped, which may be undesirable. An alternative method is to mount a small subsidiary impeller on the back of the main unit, adjacent to the gland, so that an area of low pressure is created. Neither of these precautions has given absolute satisfaction, and the most effective method is to submerge the whole pump in the liquid being pumped, as shown in Fig. I–12. The impeller rotates inside a casing which is open to the liquid, and the outlet stream is forced into a

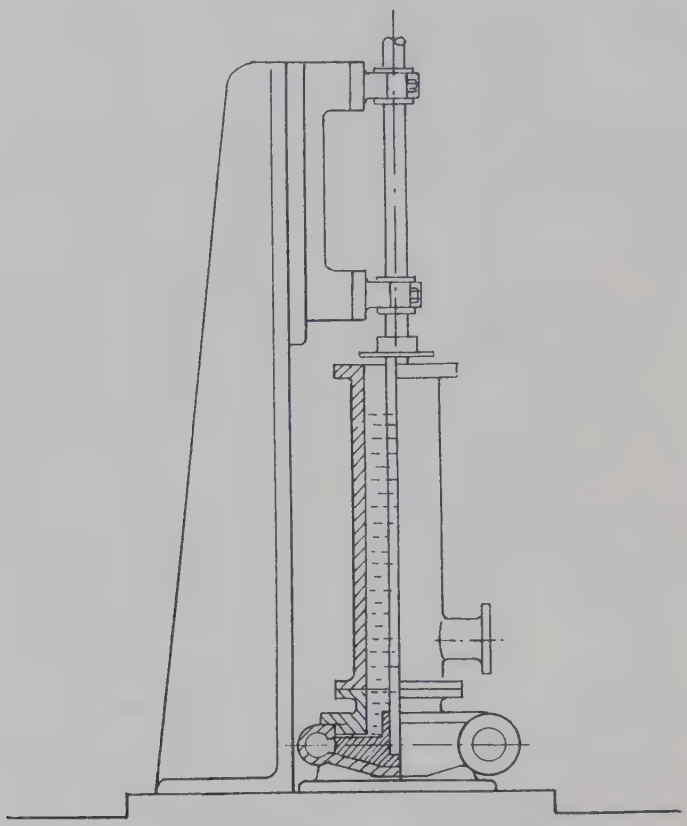


FIG. I–12.—SUBMERGED GLANDLESS CENTRIFUGAL PUMP.

suitable channel and up out of the tank ; the bearings for the shaft are well above the liquid level. This submergence may not always be possible, and it is difficult to arrange for a rotating system of this kind to balance properly at the high speeds required.

While it is true that the rotor and casing can be easily constructed out of such materials as chemical stoneware, the shaft has to be mechanically reliable and must be made from steel, or some similar metal. Although this steel shaft can be cased in stoneware, the joint between them is a source of weakness ; a special plastic resin is used, but if this fails at any point, acid will seep in and the

corrosion products will force the stoneware from the shaft, often with sufficient force to burst the impeller. The high speed necessary for all but the smallest heads calls for direct coupling to an electric motor, and this may be a disadvantage where it is desirable to keep all possible sources of fire outside a particular plant building. Finally, the centrifugal pump is very susceptible to any obstruction in the feed lines, and will not start to pump unless "primed" or

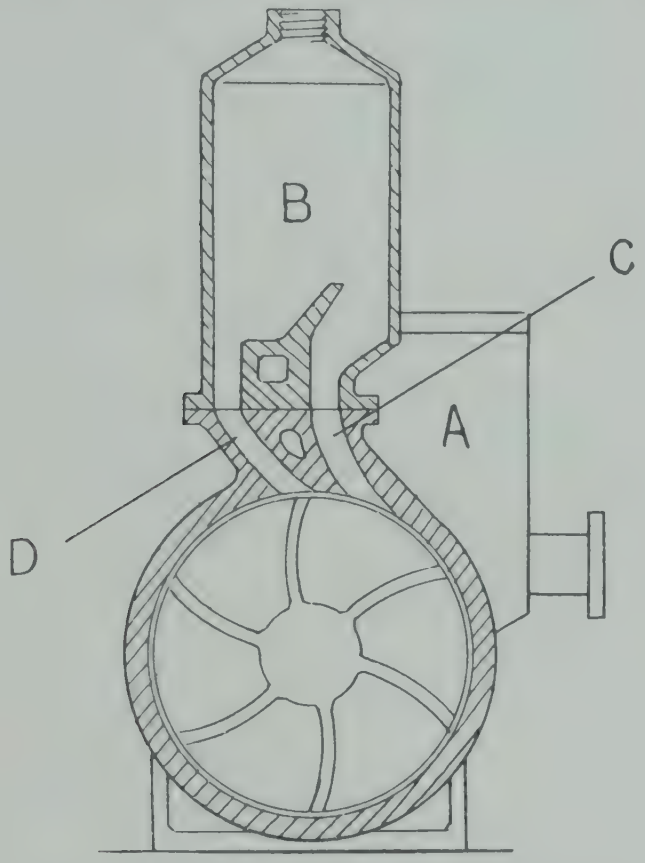


Fig. I–13.—LaBour Self-Priming Pump.
(*Courtesy of British LaBour Pump Co. Ltd.*)

filled with liquid. It is good practice to arrange for a centrifugal pump to be mounted below the level of liquid in the feed tank, so that this priming can easily be obtained, while the feed line should be as short as possible, especially where hot liquids are being handled. A number of foot-piece valves and self-priming devices are available where it is not possible to make this arrangement. The LaBour self-priming pump shown in Fig. I–13 is typical of these latter; a tank A on the suction side has a capacity not less than the separating tank B on the discharge side, and A is normally filled with

liquid when the pump is stationary. When the pump starts, this liquid enters the pump, and a mixture of air and liquid is pumped to the separating tank through outlet C. The air escapes at the top of the separator, while the liquid runs back to the pump casing through D, and this process continues until all the air in the system is discharged and the pump has taken up its normal working cycle.

The shortcomings of both reciprocating and centrifugal pumps have been a challenge to the ingenuity of inventors, and the number of peculiar pumps functioning in some intermediate fashion is very large. The two principal classes are based upon the gear pump (Fig. I–14a) and the eccentric-cylinder type (Fig. I–14b). In the

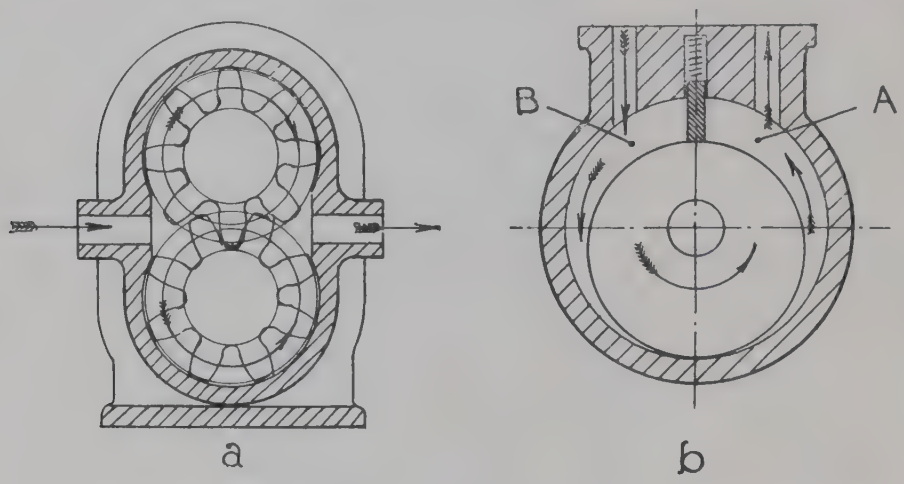
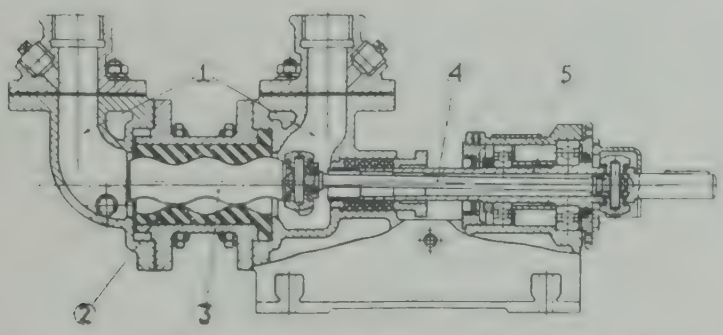


Fig. I–14.—Rotary Pump Types.

gear pump two gears mesh closely at the centre, and as the wheels rotate, the space between the teeth increases on the one side, while it diminishes on the other as the teeth enter into mesh. The fluid being pumped will enter on the former side, travel round the periphery of the gear case, and be forced out on the discharge side as the gear teeth mesh. In the rotary pump of Fig. I –14b, the volume represented by area A is decreasing, while that represented by area B is growing, with the result that a steady stream of liquid is drawn in at the right-hand port and discharged at the left-hand. The gear pump may actually be used in a form almost identical with the simple outline of Fig. I 14a, but the eccentric-cylinder forms are usually more complicated. Both types work at speeds which may vary over a wide range, but are usually within the limits of 250–1000 r.p.m.

These pumps depend for their efficiency on closely machined clearances between two metal surfaces. If a self-lubricating liquid, such as oil, soap solution, or even certain salt solutions is being pumped, satisfactory service over a period of years may be obtained. On the other hand, a gritty or corrosive liquid may wreck the pump completely in a few hours. Rotary pumps should never be used, therefore, in pumping chemicals which have even a slight corrosive effect on the material of construction. They are cheap—about the same cost as centrifugal units for throughputs of below 1000 gal./hr.—and can be made to give fluid pressures up to 2000 lb./sq. in. The discharge rate is proportional to the pump speed, but the pressure is almost independent, although there is a tendency for



1. Suction and Delivery branches. By reversing rotation, direction of flow can be arranged either Suction or Delivery on Gland.
2. Stator. 3. Rotor. 4. Universal Drive to rotor.
5. Driving shaft, running in ball bearings.

FIG. I–15.—"MONO" PUMP.

(*Courtesy of Monopumps, Ltd.*)

output to fall as the pressure increases, owing to seepage past the close-fitting surfaces. This is especially marked with the simpler types of pump, which are seldom used at outlet pressures exceeding 100 lb./sq. in.

Modern pump designs have attempted to overcome this drawback, and also the tendency shown, in common with centrifugal pumps, to develop leaks at the glands carrying the shafts. The simple eccentric cylinder can be extended into a spiral rotor, moving in a grooved casing, as in the "Mono" pump of Fig. I 15. The rotor is of metal and the casing of rubber, and this pump will handle solid-liquid suspensions and work to pressures of the order of 100 lb. sq. in. without requiring any special priming devices. An

ingenious combination of slide-valve action combined with eccentric rotary motion is found in the "Megator" pump (Fig. I–16), where three eccentric discs rotate freely in hard rubber "shoes," which in turn slide up and down against a cover plate, closing or opening discharge ports to engage with the motion of the rotors. This pump also will give a steady discharge of solid–liquid slurry at pressures up to 100 lb./sq. in. These two pumps have been chosen almost at random from a very large number of similar devices, all based on the eccentric-cylinder principle.

The single-gear pump has the advantage of simplicity, and is best used with viscous liquids, which cut down the slip past the blades. A unit of this type, pumping fruit syrup, worked at 250

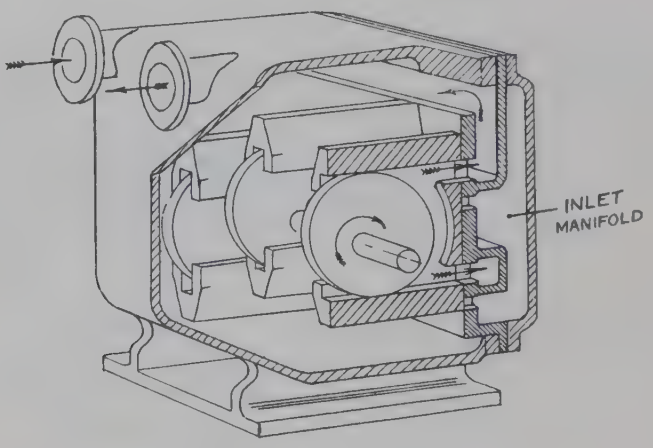


FIG. I–16.—"MEGATOR" PUMP.
(*Courtesy of Megator Pumps, Ltd.*)

r.p.m. with a volumetric efficiency of about 60%, at a discharge pressure of 20 lb./sq. in. If high pressures are required, the gear can be lengthened to a screw or double screw. In a typical instance two shafts carry right- and left-hand threads which intermesh and force a liquid towards a discharge in the centre of the casing; this can be used as a boiler feed pump, forcing liquid into boilers against pressures of more than 1000 lb./sq. in.

One special type of pumping, used in chemical works for the circulation of relatively small quantities of corrosive liquids, remains to be mentioned. This is the air-lift, a diagram of which is shown in Fig. I–17. As will be seen, it is a U-shaped tube with one limb considerably longer than the other; at the base of this limb, compressed air is delivered at a pressure slightly greater than the fluid

pressure. The air mixes evenly with the liquid as it flows through the mixing holes of the foot-piece, and builds up in the rising limb a liquid-air mixture which is lighter than the liquid filling the shorter leg of the U-tube. A steady discharge of the mixture at the top of the lift is obtained. It is very easy to improvise an air-lift, but not so easy to obtain one which is efficient. According to Swindin,* a good air-lift should have an efficiency, calculated on power fed to air-compressor, of 30–40%. In order to achieve such efficiencies, the ratio of submergence (h_s in Fig. I–17) to lift H should not be less than unity, while a balance must be made between high velocity in the lift tube and high slippage between the air bubbles and the liquid. The foot-piece in which aeration occurs should allow for a gradual increase in velocity, as otherwise entry losses can be considerable. One of the most frequent uses for these air-lifts in chemical works has been the circulation of dilute nitric acid round NO_2 absorption towers; a large excess of air did not matter, as the oxygen was needed for the oxidation of the NO_2. As a result, a clumsy stoneware foot-piece and a glass tube lift which seemed too large for the amount of liquid being handled were a commonplace in many acid works. These lifts are now being replaced by stainless steel centrifugal pumps, with independent aeration, but it might have been possible to improve the air-lift design to give better results.

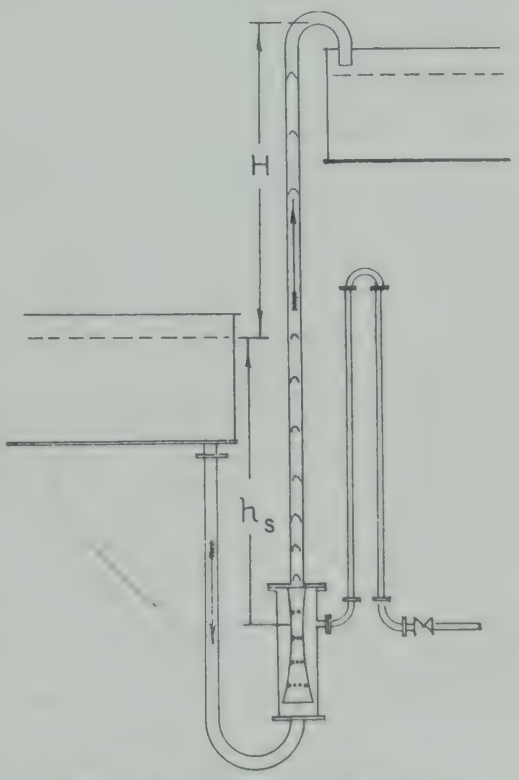


Fig. I–17. Air-Lift Pump.

No formulae have been published for very short air-lifts, but where they have been used in pumping liquid from mines—a rather different field—it has been suggested that the velocity of an air-water

* *Proc. Chem. Eng. Group*, 1928, 10, 116.

mixture should be about 10 ft./sec., and the amount of air required per gallon of water is given by the formula

$$v \text{ (cu. ft. of free air)} = 0{\cdot}8 \frac{H}{240 \log \frac{h_s + 34}{34}}$$

The air-lift is not a jet entrainment device, and makes no use of air momentum.

Jet pumps, or ejectors, in which a jet of fluid at high velocity entrains a certain proportion of another fluid, are relatively unimportant in liquid pumping. A "blow jack" or simple steam ejector can be used as a simple means for emptying sumps or drainage tanks, but the liquid is diluted by the condensed steam and the pump is generally very inefficient. The automatic boiler-feed injector pump is a much more complicated device and is not within the scope of the present work. The most important uses of entrainment pumps are in gas pumping and will be dealt with below.

Gas Pumping. Gas pumps may be of reciprocating, centrifugal, or rotary types, as in the case of liquids, and these three types are used in the same way, with the centrifugal pump giving high throughputs with low pressure differences, and the reciprocating pumps the highest pressures. Where the pressure increase through the pumping system is small, it is possible to consider gas pumping as a normal case of fluid movement, and to calculate work required on the basis of volume pumped and pressure increase. When the pressure increase is more than 2–3 lb./sq. in., this simplification is no longer possible, and the work done on the gas in compression, and resulting in volume change, must be taken into account.

In the first and simpler case, centrifugal pumps for gases take the form of the propellor or paddle-wheel fan. These have the advantage of simplicity, and can be made up in corrosion-resisting material. The propellor fan, as shown in Fig. I–18a, is primarily designed for moving large quantities of gas at very low pressure differences of not more than 1 in. of water gauge (1 in. w.g. = 0·036 lb./sq. in.). These fans are used for ventilating buildings rather than for definite movement of gases in a plant, but they have been used in the circulation of air in large drying chambers. Typical figures for propellor fans are as follows :

3 ft. diam.	425 r.p.m.	11,000 cu. ft./min.
5 ft. diam.	254 r.p.m.	30,000 cu. ft./min.

The mechanical efficiency of these fans is at a maximum of about 50% when working against a back-pressure of 0·25 in. w.g. Generally they follow the rule of all centrifugal pumps that the power required varies as the cube of the speed, but output is based on empirical test figures. Paddle-wheel fans, as shown in Fig. I–18b, are much closer to true centrifugal pumps, discharge varying with speed and pressure with the square of the speed. As gases vary widely in density, the performance of the fan will also vary widely, and for a given speed, discharge pressure and power requirements will be determined by the mean density of the gas being pumped. Any paddle-wheel fan will give characteristic curves similar to those of a liquid centrifugal pump, and will have a limiting pressure for

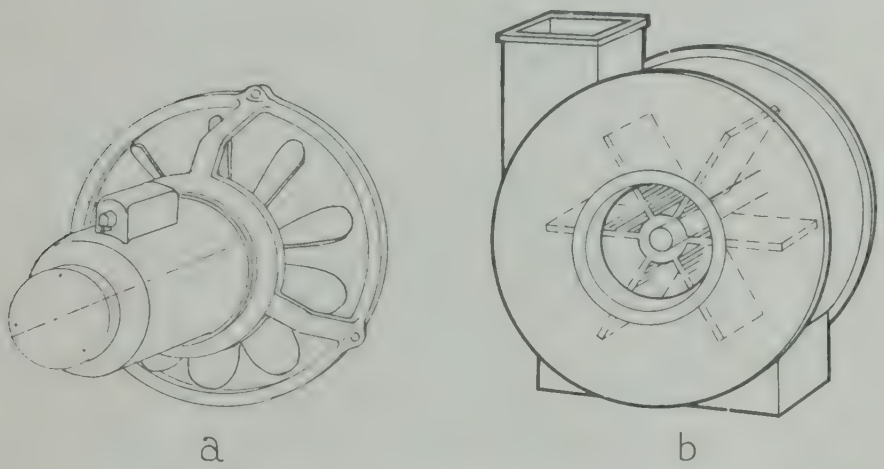


Fig. I–18.—Propeller and Paddle-Wheel Fans.

any speed, above which discharge ceases completely. This limiting pressure is set in practice at about 10 in. w.g., although special forms of fan have been built to work up to as much as 25 in. The casing of the fan closes more completely around the rotor as the designed pressure of the fan increases. These paddle-wheel units are the most common gas-handling appliances when the gas to be pumped is corrosive: a fan of stoneware, with a 16-in. rotor, has been built to give 250 cu. ft. min. of gas containing HNO_3 and HCl, at a pressure of 6 in. w.g. and a mechanical efficiency of 50%. A stainless steel unit 64 in. in diam. pumps 30,000 cu. ft. min. to 12 in. pressure for recovery of nitrous fumes. The pumps can also be used for circulating dust-laden air (as in the domestic vacuum cleaner), and have only one apparent drawback—the characteristic high-pitched whine which is heard in operation. This noise is a source of great

annoyance when working in a confined space, and it is best to arrange for the fans to be mounted outside a building.

If the desired pressure is more than 10 in. w.g., some type of rotary blower is usually employed. These blowers are the analogue of rotary liquid pumps, and are similar in principle if not in design. The gear pump is altered to the cycloidal blower shown in Fig. I–19; the dumbbell-shaped impellers sweep gas round the periphery of the casing just as the gears carry liquid. Volumetric efficiency depends upon the closeness of the fit at centre and sides, but a well made blower of this type will give up to 90% of the theoretical

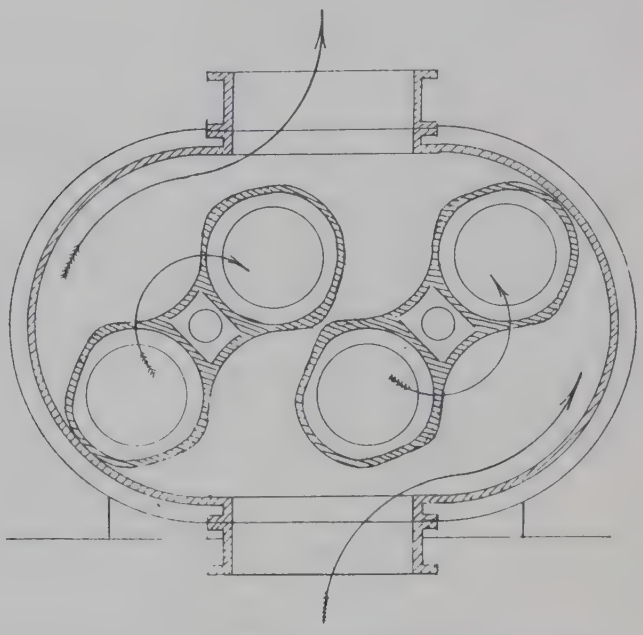


FIG. I–19.—HOLMES CONNERSVILLE BLOWER.
(*Courtesy of W. C. Holmes & Co.*)

displacement figure. In fact, a specially close-fitting double-lobe impeller unit is used as a gas meter. The rate of rotation varies from 200–400 r.p.m., and the best working pressure difference is from 1–3 lb./sq. in. Tar-laden gases from coal carbonisation can be pumped by these blowers with a mechanical efficiency of some 60%, and are more compact than the earlier multi-bladed exhausters devised for this duty. These exhausters, as shown in Fig. I–20, are clearly based on the eccentric-cylinder principle, but the design details are different. The blades are hinged independently on a central axle, and are swept round by the smaller eccentric drum. Sliding ports, of ball and socket section, allow the blades to move in

and out of the inner drum, maintaining a sliding friction fit against the outer casing. The speed of these blade tips is not more than 700 ft. per minute and the wear is small. These exhausters gave satisfactory service over long periods but were large and unwieldly: they have now been generally replaced, for large loads, by turbo-exhausters. These are highly specialised centrifugal pumps, built for loads of over 1000 cu. ft./min., and are restricted to gases of relatively non-corrosive character. A simpler exhauster, of the double-lobe type, is preferable for the smaller quantities of corrosive gases to be handled in chemical plant.

For pressures above 3 lb./sq. in. the problem is one of gas compression. As with liquids when high pressures are required, a

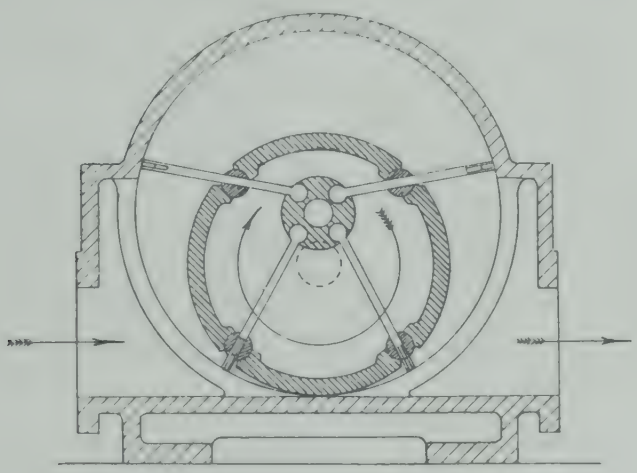


FIG. I–20.—MULTI-BLADE GAS EXHAUSTER.

reciprocating pump is usually installed. The volume changes with a gas are so great that the work done, reappearing as heat in the compressed gas, makes it vital to cool the compressor, and for pressures above four atmospheres, to compress in stages, with cooling between each stage. A typical four-stage gas-compressor is shown in Fig. I-21: it is designed to compress hydrogen in four stages from normal pressures up to 200 atmospheres. The valves are light steel rings having as little inertia as possible, while the clearance between piston and cylinder-head is kept to the smallest working limit.

The work done in the isothermal compression of a gas is given by

$$W \text{ (work in ft.–lb.)} = p_1 v_1 \log_e \frac{p_2}{p_1}$$

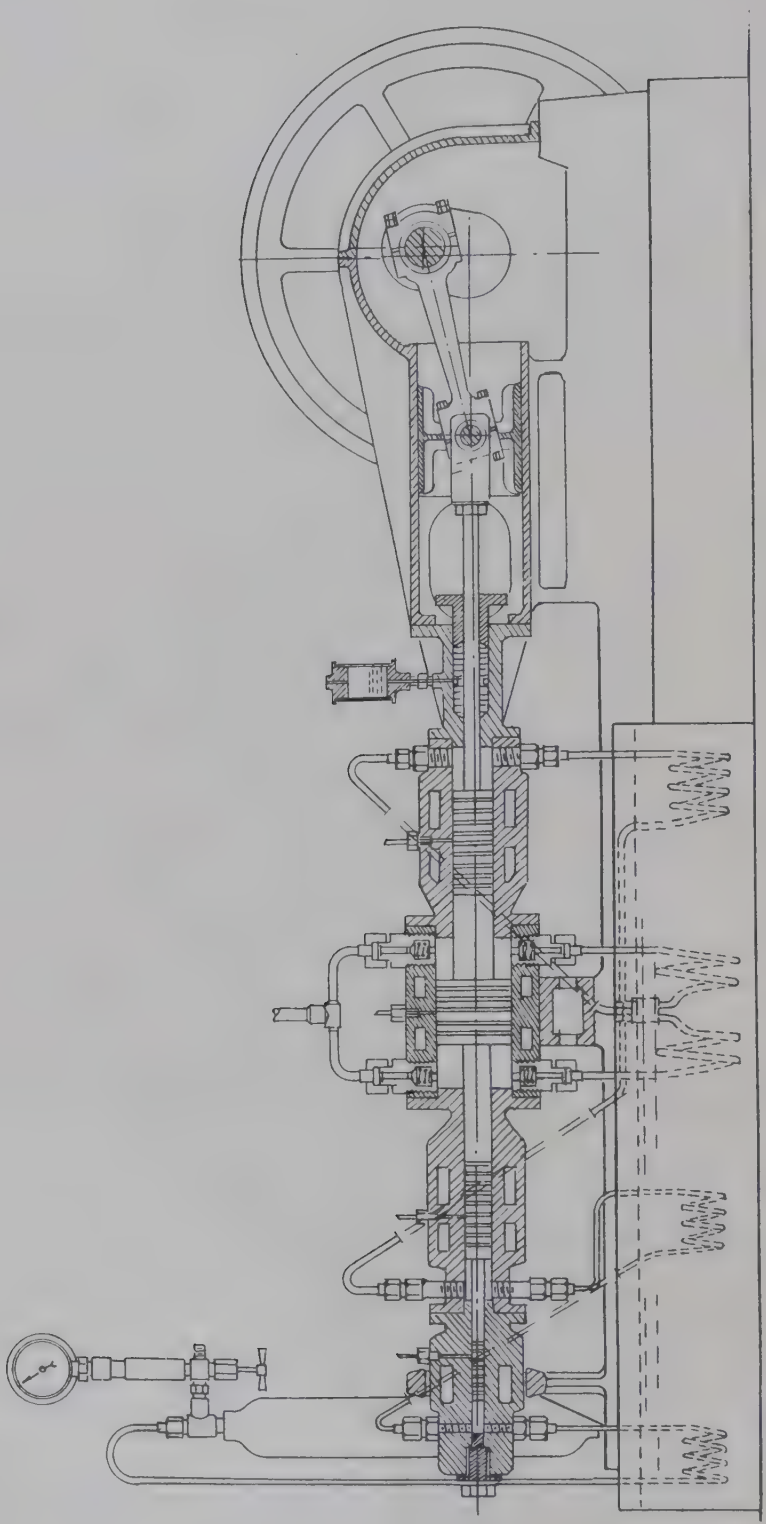


FIG. I-21.—FOUR-STAGE GAS-COMPRESSOR.

where $p_1 - c_1$ and $p_2 - c_2$ are the initial and final states of the gas being compressed (both in foot-pound units). Unfortunately for efficiency, this ideal isothermal state can never be reached, and compression always tends to draw nearer to adiabatic working, when the work required becomes

$$W \text{ (work in ft.-lb.)} = \frac{\gamma}{\gamma - 1} p_1 c_1 \left\{ \left(\frac{p_2}{p_1} \right)^{\frac{\gamma - 1}{\gamma}} - 1 \right\}$$

where γ is the ratio of specific heats. This is always greater than the work for isothermal compression, as will be seen from the table below giving the ratio isothermal adiabatic work (I/A) for various pressure ratios of air.

p_2/p_1	1·5	2·0	3·0	4·0	6·0	8·0	10
I/A	0·94	0·90	0·85	0·83	0·78	0·73	0·70

The figures above are for single-stage compression, and multi-stage working improves the adiabatic figures. Normal compression efficiency is not more than 50–60%, based on isothermal compression figures. The reciprocating compressor is always used in practice for pressures above 100 lb. sq. in., and multi-stage rotary and centrifugal pumps are not serious competitors, as in the case of liquid pumping. Whether this will always be the case is doubtful, as the efficiency of turbo-compressors of various types is growing and very large loads are becoming more common.

The wide use of reduced pressures in chemical plant has resulted in great improvement in the design of vacuum pumps of all types, from the simple reciprocating pump working down to about 5 cm. of mercury pressure, to the vapour diffusion pump which will work down to 10^{-10} mm. Hg. The first unit is merely an ordinary air-compressor discharging to atmosphere, and many pumps are used for the double duty. This is not really desirable, as the vacuum pump in a specially built form is lighter than the compressor, with a still smaller clearance volume and slighter valves. The clearance determines, of course, the limiting ratio between suction and discharge pressures: a ratio of clearance volume to swept volume of 0·01 will give a limiting vacuum of 1/100 atm. (more precisely 0·01/1·01 atm.) when exhausting to atmosphere. In practice it is general to design pumps with a somewhat larger clearance than this, and to arrange for two stages of exhaustion, as shown in the pump of Fig. I 22. The reciprocating vacuum pump works at medium speeds—piston speeds up to 500 ft. per minute—and is generally

less noisy than a rotary, multi-blade exhauster, which is often used instead. A rotary vacuum pump for pressures down to some 5 cm. Hg. (single-stage) or 5 mm. (two-stage) is a rotary pump of the eccentric-cylinder type. The multi-bladed impeller (Fig. I-23a) spins at rates up to 2000 r.p.m., and the high potential wear on the blade tips is checked by introducing a slip-ring casing which takes up the running stress. A pump of this kind will run for many years with little drop in efficiency, and is much less costly and cumber-

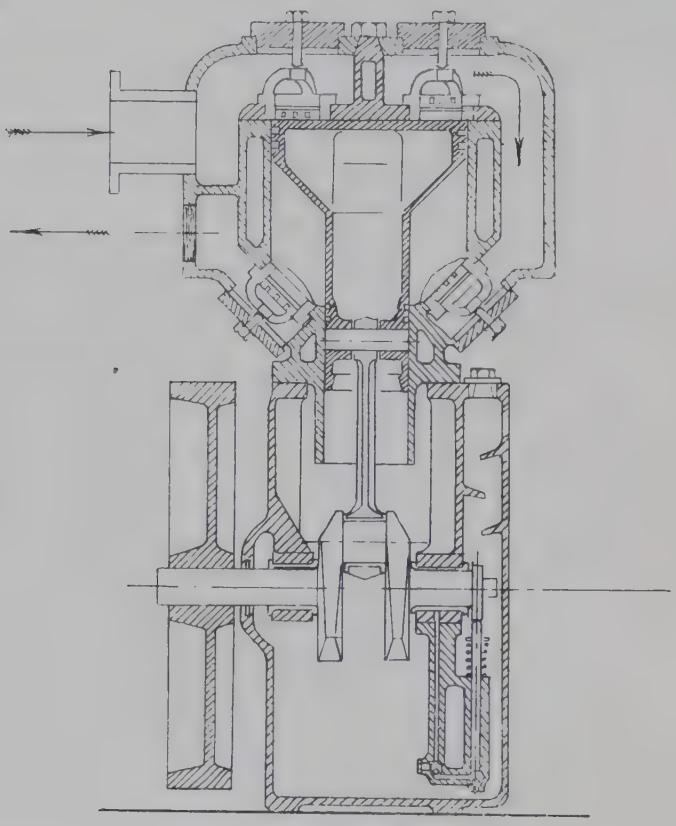


FIG. I–22.—RECIPROCATING VACUUM PUMP.
(Courtesy of Worthington-Simpson Ltd)

some than a reciprocating pump for the same duty. It is, however, very noisy, and needs a special housing if annoyance is to be avoided. The forced lubrication covers the metal surfaces with a skin of oil, and appears to check corrosion very well, but a high-speed machine of this kind needs a good constructional material. These rotary exhausters can be used for compressors up to pressures of about 30–40 lb./sq. in.

High-vacuum pumps, designed to reduce pressures to below 0·01 mm. Hg, are again of the eccentric-cylinder type, but of

different design to the high-speed exhausters of Fig. I 23a. The oil-sealed vacuum pump shown in Fig. I 23b has only two blades on the rotor, and the whole pump is immersed in oil. Provision is

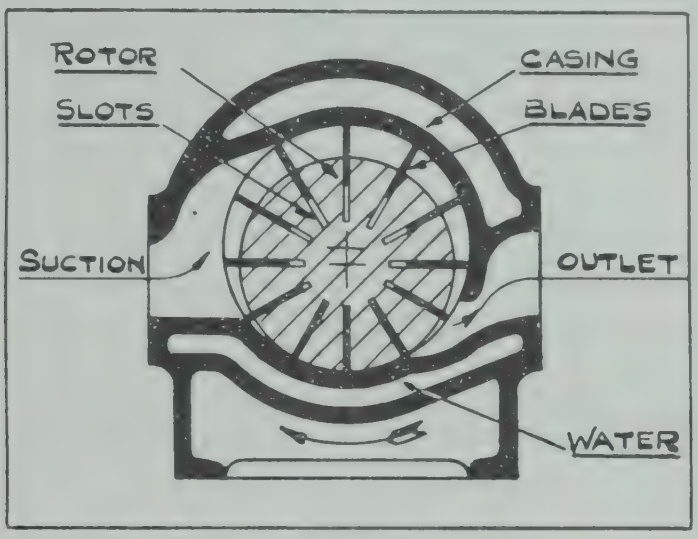


Fig. I–23a.—Rotary Vacuum Pump.

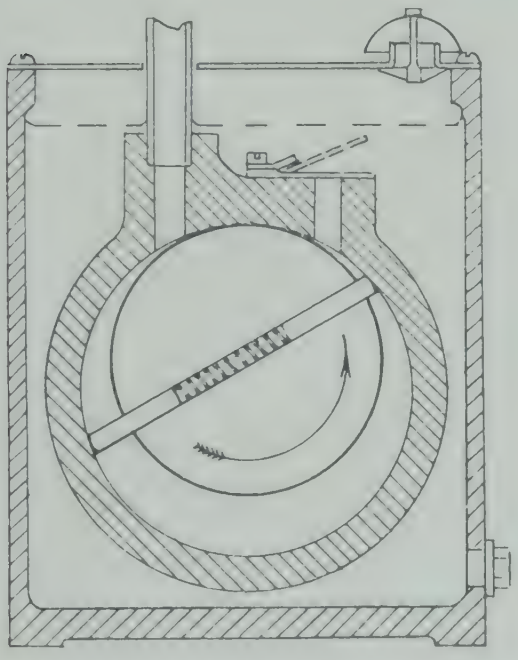


Fig. I–23b.—Oil-sealed High-Vacuum Pump.

made for oil to drain in and out of the pumping chamber, so that the rotor is discharging a mixture of oil and gas. Despite this arrangement for an oil-seal, the operation of the pump depends entirely upon accurate machining of the rotor and casing, especially

at the top and ends. The seal between high and low pressure is made at these points, and very slight flaws are fatal to efficiency. As a result it is essential that no corrosive or abrasive matter shall enter these pumps, and they must be provided with effective traps. Mercury, so often associated with vacuum work, makes a very efficient abrasive when mixed with oil. The quality of the oil in the pumps is also important, as traces of moisture or low-boiling constituents make the pump quite useless for pressures below 1 mm. or so. It is difficult to assess the efficiency of these pumps on a mechanical basis, as the important factor is the end vacuum obtainable.

The high cost and potential rapid wear of these rotary pumps has led to the extension of steam-jet vacuum pumping into these high-vacuum fields. An ordinary steam-jet exhauster, working as an entrainer, will remove about 0·2 lb. of air per lb. of steam used, and give a pressure ratio between inlet air and outlet air pressures of 1 to 7. It is simple to arrange for a number of jets in series, as shown in Fig. I–24. The following table shows the end vacuum obtainable for the various stages:

Single Stage	100 mm.
Double Stage	20 mm.
Triple Stage	2·5 mm.
Quadruple Stage	0·4 mm.
Quintuple Stage	0·05 mm.

A discussion* on the performance of these steam-jet pumps suggests that they are somewhat more efficient than reciprocating vacuum pumps for their specific duty range. The jet pump can be made up in such chemically resistant material as graphite or earthenware.

The final stages of very high-vacuum work are reached by vapour-diffusion pumps, devised originally by Gaede and Langmuir. A pump of this kind is shown in Fig. I–25, and consists of a boiler A in which a stable, high-boiling liquid boils under the vacuum (below 0·1 mm. as a rule) applied by the backing pump. These diffusion pumps will not work until the vacuum is reduced to the backing pump limits. The vapour passes up a central conduit and down through the baffles C, C. The vapour stream partly entrains and partly receives by diffusion the gas molecules in the high-vacuum space D and carries them down into the condensing zone E, where the vapour condenses and the residual gas molecules are pumped

* G. Arrowsmith, *Trans. Inst. Chem. Eng.*, 1949.

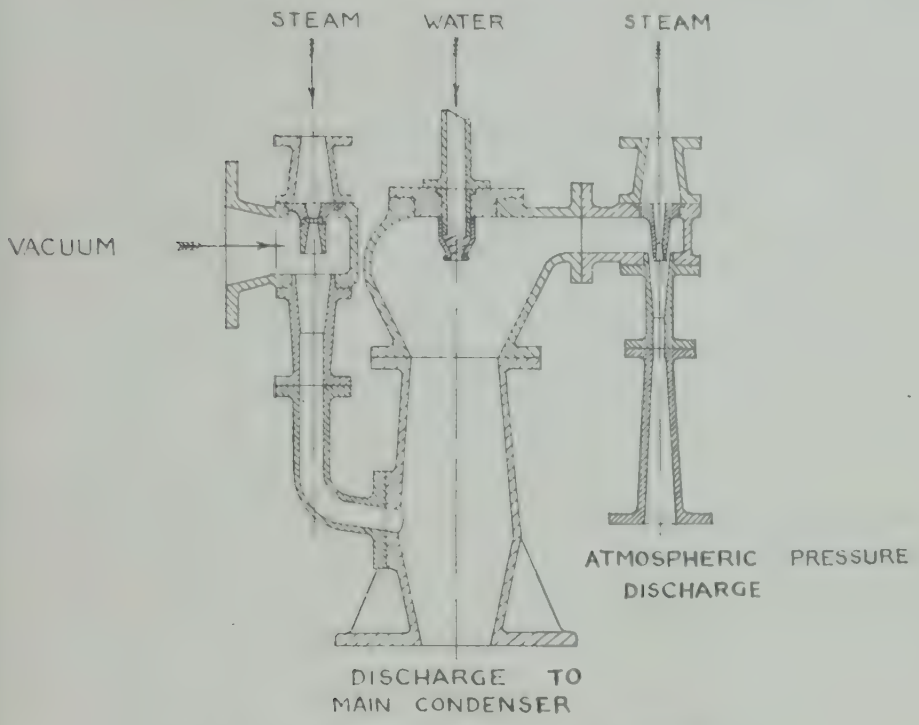


FIG. I-24.—TWO-STAGE STEAM-JET EXHAUSTER.

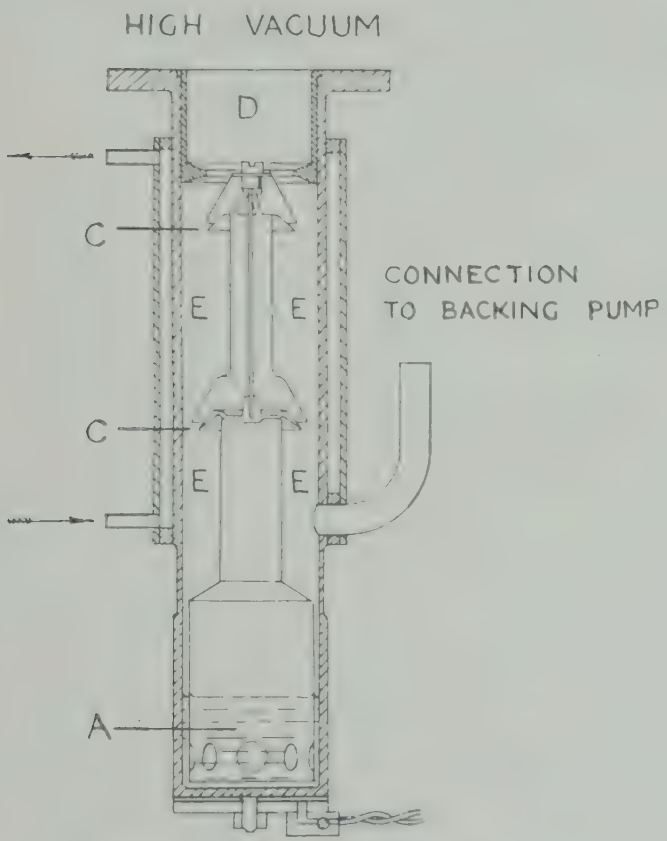


FIG. I-25. TWO-STAGE DIFFUSION PUMP.

out by the backing pump. The actual design of these diffusion pumps is still a matter of controversy, but they are capable of dealing with very large volumes of gas at low pressures. A pump with a throat 6 in. in diameter can have a pumping speed of 1200 cu. ft./min. at a pressure of 0·0001 mm. Hg (0·1 micron). Again, it is not possible to trace a relation between heat input to boiler and amount of gas exhausted from a space kept at 1 micron to a space at 100 microns, and the pump efficiency is considered by a measure of the end vacuum produced. The original high-boiling liquid was mercury, but several oils have been made up for this duty and are tending to replace mercury in commercial service.

The design of pipe-lines for vacuum practice is complicated by the very large volumes of low-pressure gas to be handled, and where pressures are as low as 1 micron or less, any sort of connecting pipe is inadmissible. The working chamber must be built on top of the pump, or the mouth of the diffusion unit must open directly into the unit. Where somewhat higher pressures are used, the problem is still complicated, as shown in a paper by Griffiths.* Several instances of the connection of plant to pumping appliances are shown by Leyland† in an account of the applications of vacuum to industrial chemical processes.

* H. Griffiths, *Trans. Inst. Chem. Eng.*, 1945, 23, 113.
† J. Leyland, *ibid.*, 1949.

CHAPTER II

HEAT TRANSFER

RATES of heat transfer are the most common predominating factor in chemical plant design and operation. For a large majority of chemical reactions carried out on an industrial scale, the limiting factor will always be the rate at which heat can be removed from, or added to, the reaction system. Heat flow may be through conduction, convection, or radiation, and is usually through a complex path with more than one resistance to flow. All three forms of heat transfer are usually concerned in any practical problem, but except at high temperatures and in the heating of gases the effect of radiation is small.

Heat flow by conduction through any material is governed by Fourier's Law, which states that the quantity of heat (Q) which is passing is determined by the equation

$$Q = - KA \frac{dT}{dL}$$

Here A is the cross-section of the heat path and dT the change in temperature over a path length dL. The constant K in the equation is defined as the thermal conductivity of the material, and is expressed, for foot-pound-second units, as B.Th.U./sq. ft./sec. F. per foot of thickness. It may, of course, have any other self-consistent form. Then the heat flow per unit area for a thickness of L ft., with a temperature difference of T F. will be $K\frac{T}{L}$. When several such material paths are arranged in series, each with the same heat flow, then

$$K_1 \frac{T_1}{L_1} = K_2 \frac{T_2}{L_2} = K_3 \frac{T_3}{L_3} \ . \ . \ .$$

and the overall temperature drop required for heat flow Q will be

$$T_1 + T_2 + T_3 + \ . \ . \ .$$

If the resistance to heat flow of a composite path of unit area is defined as the temperature drop for unit heat flow, then in the case above

$$\text{Resistance} \quad (R) = \frac{T_1 + T_2 + T_3 + \ldots}{Q}$$

$$= \frac{T_1}{Q} + \frac{T_2}{Q} + \frac{T_3}{Q} + \ldots$$

or

$$R = R_1 + R_2 + R_3 + \ldots$$

For any system the reciprocal of overall resistance, or the heat flow for unit temperature drop, may be defined as the characteristic heat transfer coefficient H. It follows that

$$\frac{1}{H_a} = \frac{1}{H_1} + \frac{1}{H_2} + \frac{1}{H_3} \ldots$$

where H_a is the overall heat transfer coefficient, and H_1, H_2, H_3, etc., the individual coefficients.

Then

$$H_a = \frac{1}{1/H_1 + 1/H_2 + 1/H_3 + \ldots}$$

(H_a may be expressed in B.Th.U./sq. ft./sec./°F. or similar units.) This expression is a more convenient form for calculation than others based on "resistances", or on thermal conductivities with the need for determination of thickness. As will be seen later, it is very difficult to estimate the thickness of a fluid film, but expression in the form of a "film" heat transfer coefficient allows of rapid computation of overall heat flow.

In the case of purely conductive heat flow, it is simple to calculate the various coefficients, and from them the overall coefficient, if the absolute conductivity values are known. A typical calculation is shown in Example 1.

Example 1. *A tank of copper, $\frac{1}{4}$ in. in wall thickness, is lagged with 2 in. of magnesia lagging and covered by 1-in. wooden slats. Calculate the overall heat transfer coefficient given that K for copper is 220, for magnesia 0·04, and for wood 0·1 (all in B.Th.U./sq. ft./hr./°F. per ft.).*

The coefficient for the $\frac{1}{4}$-in. copper wall is

$$220 \times \frac{48}{1} = 10{,}560 \text{ B.Th.U./sq. ft./hr./°F.}$$

For the 2 in. of magnesia

$$0{\cdot}04 \times \frac{12}{2} = 0{\cdot}24 \ B.Th.U./sq.\ ft./hr./^\circ F.$$

For the 1 in. of wood

$$0{\cdot}1 \times 12 = 1{\cdot}20 \ B.Th.U./sq.\ ft./hr./^\circ F.$$

Then $$H_a = \frac{1}{1/10560 + 1/0{\cdot}24 + 1/1{\cdot}20} = \frac{1}{0{\cdot}0001 + 4{\cdot}6 + 0{\cdot}83}$$
$$= 0{\cdot}18 \ B.Th.U./sq.\ ft./hr./^\circ F.$$

Clearly the copper wall has a negligible effect.

So far it has been assumed that the flow of heat has been along a uniform cross-section. In many important cases heat flow is across

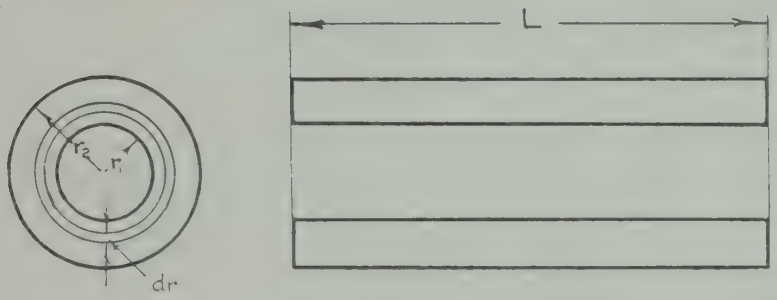


Fig. II–1.

a cylindrical body of gradually increasing cross-section, as shown in Fig. II–1. Here heat is flowing from the inner to the outer surface of a hollow cylinder. The area of any cylindrical element of thickness dr will be $2\pi rL$. Let the rate of heat flow be Q with a temperature change dT over a thickness dr. Then by definiiton

$$\frac{Q}{2\pi rL} = -K\frac{dT}{dr}, \text{ or } \frac{Q}{2\pi L}\frac{dr}{r} = -kdT$$

As Q is constant for all values of r, this equation can be integrated between the limits r_1, r_2 and T_1, T_2.

Then $$\frac{Q}{2\pi L}\log_e\frac{r_2}{r_1} = K(T_1 - T_2)$$

whence $$Q = \frac{2\pi LK}{\log_e r_2/r_1}(T_1 - T_2) \quad . \quad . \quad . \quad . \quad . \quad (1)$$

Consider a mean radius R which would give a path equivalent to the actual varying cross-section of the cylinder. Then the equivalent

area will be $2\pi LR$ with a thickness of $r_2 - r_1$ and the rate of heat flow will be

$$K2\pi LR \times \left(\frac{1}{r_2 - r_1}\right) \times (T_1 - T_2).$$

But by definition this will be equal to (1) above, and therefore

$$R = \frac{r_2 - r_1}{\log_e r_2/r_1}$$

This is the logarithmic mean of r_1 and r_2, a mean which is of great importance in problems of heat transfer.

Example 2. *Calculate the heat loss per foot run from a 6-in. steam pipe covered with 3 in. of glass wool lagging* ($K = 0{\cdot}030$) *when the surface of the pipe is at 500° F. and the outer lagging surface is at 100° F.*

Here $r_1 = 3$ *in. and* $r_2 = 6$ *in. The log mean radius is* $(6-3)/\log_e \frac{6}{3} = 4{\cdot}34$ *in. The mean area per foot run is* $2\pi \times 4{\cdot}34/12$, *or 2·27 sq. ft. The heat transfer coefficient is* $0{\cdot}03 \times \frac{12}{3} = 0{\cdot}12$.

$$\therefore \quad \textit{Heat loss} = 2{\cdot}27 \times 0{\cdot}12 \times (500 - 100)$$
$$= 109 \textit{ B.Th.U./ft. run/hour.}$$

(*K is in B.Th.U./sq. ft./hr./°F. per ft.*).

It has been assumed in this example that the value of K does not change appreciably with temperature. This is generally true, as variations with temperature are not great, and a mean value can be taken without undue error.

Heat flow by means of fluid movement—convection—is much more complicated than conduction. Convection may be " natural " through changes of density, or " forced " by stirring or pumping at high velocities. Exploration of the typical system of convective heat transfer—from fluid to fluid across a dividing solid wall—with a delicate temperature recorder reveals the temperature distribution set out in Fig. II–2. The main stream of each fluid is at a reasonably constant temperature, but adjacent to the wall rapid changes occur, indicating the existence of a more or less stagnant film. This film constitutes the main opposition to heat transfer.

The thickness of this film will be determined by the tearing action of the body of the fluid, which in turn will be a function of fluid density, velocity, and viscosity, and of the relation between the area of the surface and the dimensions of the fluid stream. These

relations may be expressed by the assumption that film thickness varies as $v^a \times \eta^b \times \rho^c \times l^d$, where ρ is the density, η the viscosity, v the velocity, and l some linear dimension of the wall. On a dimensional basis this assumes that

$$L = \left(\frac{L}{T}\right)^a \times \left(\frac{M}{LT}\right)^b \times \left(\frac{M}{L^3}\right)^c \times L^d$$

where a, b, c, and d are arbitrary constants. It follows that

$$L = L^{a-b-3c+d} \times M^{b+c} \times T^{-a-b}$$

and from this $a = -b = c$, while $d = a + 1$. The thickness will therefore vary as $(v\rho l/\eta)^a \times l$.

The importance of Reynolds' Criterion, $v\rho l/\eta$, has already been briefly indicated in the previous chapter. It is dimensionless

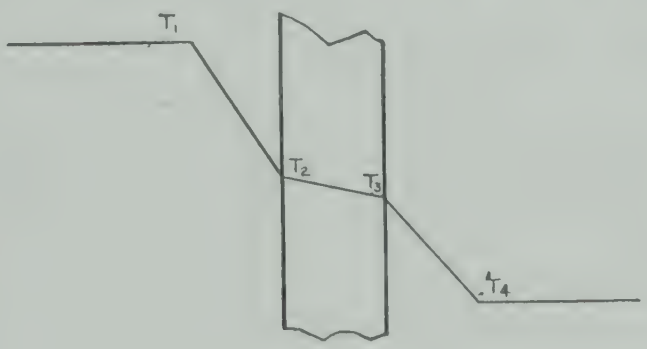


FIG. II–2.

($L/T \times M/L^3 \times L \times LT/M = 1$) and has therefore the same numerical value whatever self-consistent units are chosen as a basis for calculation. In order to compare and correlate systems of heat transfer, it is necessary to ensure that the fluid film thickness shall be either constant or vary in some regular manner; a control on this can be established by calculation of the value of Reynolds' Criterion for the system. Similar reasoning for other factors governing convective heat flow has led to the suggestion that all heat transfer coefficients (h_f) for the flow of heat from a fluid stream to a solid wall may be expressed in an equation of the form

$$\left(\frac{h_f l}{K}\right)^A = \left(\frac{v\rho l}{\eta}\right)^B \times \left(\frac{C_s \eta}{K}\right)^C$$

Here C_s is the specific heat and K the specific conductivity of a fluid of density ρ and viscosity η travelling with velocity v through a

system of characteristic dimension l. The indices A, B, and C are to be determined experimentally. This general correlation is sometimes known as the Dittus-Boelter relation, and involves three non-dimensional groups, the quantities inside the brackets. These are denoted as the Nusselt ($h_f l/K$), the Reynolds, and the Prandtl Numbers of the fluid system.

The basis of the theoretical analysis has been an assumption that the fluid is in such a state of turbulence due to forced convection that thermal or natural convection is of little importance. Thus no term for temperature difference appears in the correlation. In practice this is almost invariably the case, as all heat transfer equipment is designed to give as high a degree of turbulence as is practically possible. The degree of turbulence can be estimated from the value of the Reynolds Number. In the specific case of flow inside a cylindrical pipe, the characteristic linear dimension becomes d, the diameter of the pipe; as shown in the section on pumping. the value of Reynolds Number (Re) must exceed 2000–3000 if flow is to be turbulent at all. For conduits of non-circular section, it is generally accepted that $4m$, where m is the hydraulic mean depth, can be substituted for d with a similar range of values for change from viscous to turbulent flow. If flow is viscous, the general correlation no longer applies, though special forms with a term involving temperature difference have been suggested. Where considerable changes occur in the properties of the fluid over the temperature range in which heat flow is taking place, it may be difficult to choose a correct value for substitution in the characteristic equation. The general form of equation is also inapplicable where condensation or vaporisation is occurring at the surface, but with these reservations it is possible to draw up general "non-dimensional" equations for film coefficients which cover a very wide field of operating conditions.

The best substantiated form is in the case of turbulent flow inside a cylindrical pipe, when l becomes the pipe diameter. Then, for fluid being heated,

$$h_f = 0{\cdot}023 \frac{K}{d} \left(\frac{vd\rho}{\eta}\right)^{0{\cdot}8} \left(\frac{c_s \eta}{K}\right)^{0{\cdot}4}$$

while for fluid being cooled

$$h_f = 0{\cdot}023 \frac{K}{d} \left(\frac{vd\rho}{\eta}\right)^{0{\cdot}8} \left(\frac{c_s \eta}{K}\right)^{0{\cdot}3}$$

These equations may be applied with reasonable accuracy ($\pm 20\%$) whatever the nature of the working fluid.

The same equations may be used for a fluid passing outside a circular pipe, along the length of the tube, with a suitable substitution for d based on the hydraulic mean depth of the outer conduit.

Example 3. *Determine the overall heat transfer coefficient in a heat exchanger made up from concentric tubes of 2 in. and 1 in. internal diam., with an inner tube wall thickness of 0·05 in. of copper. The two liquids flowing are* (1) *cooling from 90° to 50° in the inner tube,* (2) *heating from 20° to 60° in the outer tube, and both are flowing at 200 gal./hr. The physical properties of the liquids may be taken as those of water at the same temperatures.*

From tables K will be approximately $7{\cdot}4 \times 10^{-5}$ *throughout,* $c_s = 1$ *and* $\rho = 62{\cdot}3$ *lb./cu. ft. (all in F.P.S. units).*

(*a*) *Inner Tube. The linear velocity given by 200 gal.* (32 *cu. ft.*) *in a 1-in. bore tube is 1·65 ft./sec.*

Then
$$\frac{vd\rho}{\eta} = \frac{1{\cdot}65 \times 0{\cdot}0835 \times 62{\cdot}3}{2{\cdot}9 \times 10^{-4}} = 29{,}600$$

(*The value for* η *is a mean figure for the 50°–90° range.*)

$$\left(\frac{vd\rho}{\eta}\right)^{0{\cdot}8} = 3758$$

$$\left(\frac{\eta c_s}{K}\right)^{0{\cdot}3} = \left(\frac{2{\cdot}9 \times 10^{-4} \times 1}{7{\cdot}4 \times 10^{-5}}\right)^{0{\cdot}3} = (3{\cdot}92)^{0{\cdot}3} = 1{\cdot}51$$

$$\frac{K}{d} = \frac{7{\cdot}4 \times 10^{-5}}{8{\cdot}35 \times 10^{-2}} = 8{\cdot}85 \times 10^{-4}$$

$$\therefore \quad h_f = 0{\cdot}023 \times (8{\cdot}85 \times 10^{-4}) \times 3{\cdot}758 \times 10^{3} \times 1{\cdot}51$$

$$= 1{\cdot}15 \times 10^{-1}\ B.Th.U./sq.ft./sec./°F.$$

(*b*) *Outer Tube. The wetted perimeter* (*two pipe surfaces*) *is 0·811 ft. and the cross-section* $1{\cdot}52 \times 10^{-2}$. *The hydraulic mean depth is* $\frac{1{\cdot}52 \times 10^{-2}}{8{\cdot}11 \times 10^{-1}}$ *or* $1{\cdot}88 \times 10^{-2}$, *giving 1in. the "equivalent diameter", as* $7{\cdot}5 \times 10^{-2}$. *The fluid velocity will be 0·59 ft./sec.*

Then
$$\frac{v4m\rho}{\eta} = \frac{0{\cdot}59 \times 7{\cdot}5 \times 10^{-2} \times 62{\cdot}3}{5{\cdot}0 \times 10^{-4}} = 5550$$
$$(5550)^{0{\cdot}8} = 1000$$
$$\left(\frac{\eta c}{K}\right)^{0{\cdot}4} = \left(\frac{5{\cdot}0 \times 10^{-4}}{7{\cdot}4 \times 10^{-5}}\right)^{0{\cdot}4} = (6{\cdot}76)^{0{\cdot}4} = 2{\cdot}15$$
$$\frac{K}{4m} = \frac{7{\cdot}4 \times 10^{-5}}{7{\cdot}5 \times 10^{-2}} = 9{\cdot}87 \times 10^{-4}$$
$$h_{f2} = (9{\cdot}87 \times 10^{-4}) \times (1000) \times (2{\cdot}15) \times 0{\cdot}023$$
$$= 4{\cdot}9 \times 10^{-2}\ \textit{B.Th.U./sq. ft./sec./°F.}$$

The copper wall has a specific conductivity of 220 *B.Th.U./sq. ft./hr./°F. per foot, or* 14·6 *B.Th.U./sq. ft./sec./°F. per* 0·05 *in.*

$$H_a = \frac{1}{1/0{\cdot}115 + 1/0.049 + 1/14{\cdot}6} = \frac{1}{8{\cdot}7 + 20{\cdot}4 + 0{\cdot}07}$$
$$= 3{\cdot}4 \times 10^{-2}$$

In the more general terms of B.Th.U./sq. ft./hr./°F., H_a becomes 123. *It will be noted that the effect of the metal wall on heat transfer is almost negligible. For strict accuracy a correction should be made for the difference between inner and outer surface area, but the effect is small and the error usually well within the accuracy of the calculated figure.*

The concentric pipe heat interchanger for which this calculation was carried out is very common in chemical plants, and a typical

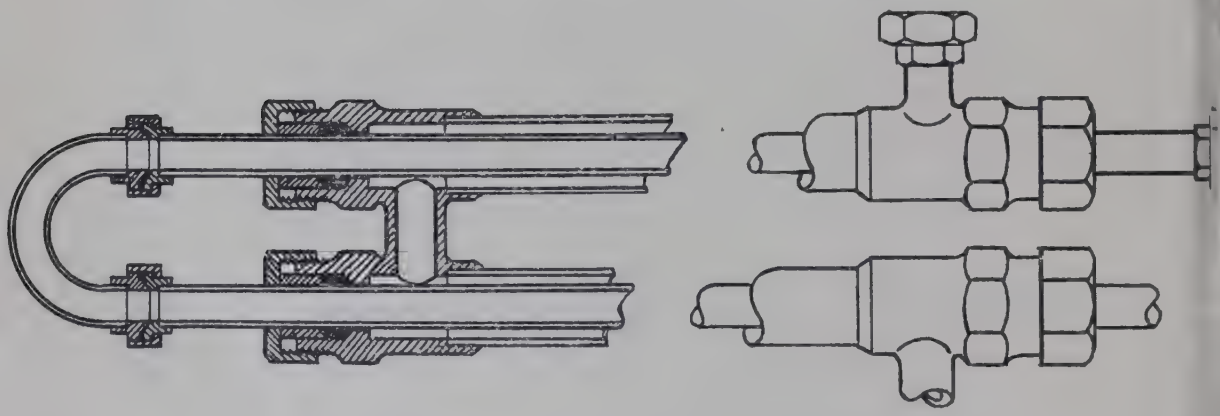


Fig. II–3.—Concentric Tube Heat Exchanger.

construction is shown in Fig. II–3. As a rule these exchangers are used for relatively small loads—up to 1000 gallons per hour, or for very corrosive materials. For these latter, the inner tube can be

made of, e.g., stainless steel or resin-bonded graphite ; the values of K for these materials are respectively 16 and 60* (B.Th.U./sq. ft./hr./°F. per ft.). If a 0·05-in. wall of the first, or a 0·2-in. wall of the latter, were used in the heat interchanger of the example above, the value of the wall coefficient would be about 1 instead of 14·6, but the change in the value of the overall coefficient would be from $3{\cdot}4$ to $3{\cdot}3 \times 10^{-2}$, a change which is well within the limits of experimental error. For large quantities of less corrosive chemicals a tube and shell heat interchanger is cheaper and more compact. In this type of plant, as shown in Fig. II-4, the outer fluid moves over a bundle of tubes mounted in tube plates at each end. The flow of the liquid in these pipes may be either once through—

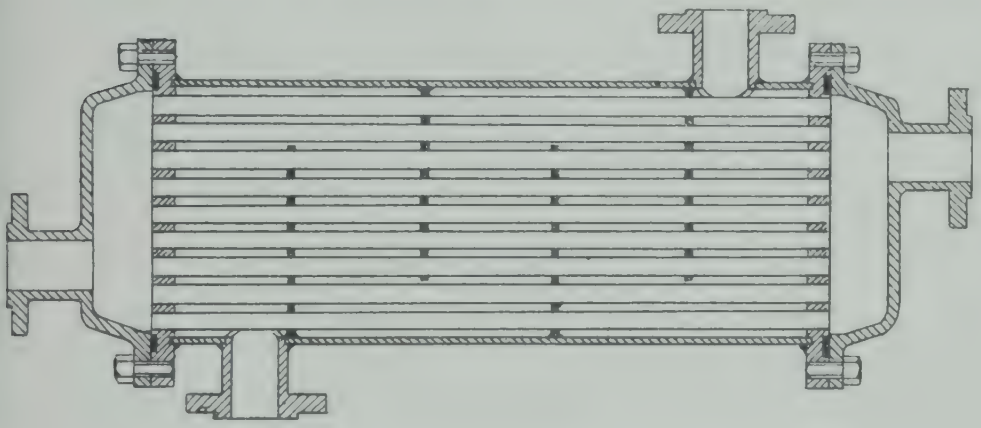


Fig. II–4.—Tube-and-Shell Heat Exchanger.

" single pass "—or backwards and forwards for any desirable number of " passes ". These inner tubes are almost always used for liquids, and gases or vapours flow on the " shell " side, with baffle plates arranged to check short-circuiting from inlet to outlet. The outer fluid will therefore pass at right angles to the tube length, and a general equation for this case is as follows:

$$h_f = 0{\cdot}385 \frac{K}{d} \left(\frac{c_s \eta}{K}\right)^{0{\cdot}3} \left(\frac{vd\rho}{\eta}\right)^{0{\cdot}56}$$

This is for a single tube of diameter d, and for tube bundles, arranged in staggered rows, the single tube coefficient must be multiplied by a factor of 1·25. The experimental value of Reynolds Number for which flow appears to change to turbulent conditions is as low as 100, but the irregular form of the fluid path would account for this.

* Hatfield and Ford, *Trans. Amer. Inst. Chem. Eng.*, 1946, 42, 121.

Alternative assumptions may be made for the method of flow on the shell side, but this hypothesis of flow normal to the pipes seems the most reasonable.

Example 4. *A tube and shell heat exchanger of the type shown in Fig.* II 4 *has* 1-*in. internal diameter copper pipes of* 0·05-*in. wall, each carrying* 200 *gall. of water per hour. This water is to be cooled from* 90°–50°. *The outer shell is* 12 *in. in diam. and carries baffles* 12 *in. apart, while the tube bundle has* 6 *tubes at the mid-section. The liquid in the outer shell is also water, flowing at a rate of* 6000 *gal./hr., and heating from* 20° *to* 60° *C. Calculate the overall heat transfer coefficient.*

A value for the inner film coefficient can be taken from Example 3—$1{\cdot}15 \times 10^{-1}$ *B.Th.U./sq. ft./sec./°F. Velocity of the shell fluid will be a minimum at the mid-section; the total path area will be* 1 *ft. by* 1 *ft., but the projected tube area is* 0·55 *sq. ft.* ($6 \times 0{\cdot}092$), *giving a total free path of* 0·45 *sq. ft. This, for a liquid volume of* 0·267 *cu. ft./sec., means a linear velocity of* 0·595 *ft./sec.*

$$\left(\frac{vd\rho}{\eta}\right)^{0\cdot56} = \left(\frac{0{\cdot}595 \times 9{\cdot}2 \times 10^{-2} \times 62{\cdot}3}{5{\cdot}0 \times 10^{-4}}\right)^{0\cdot56}$$

$$= (6830)^{0\cdot56} = 143$$

$$\left(\frac{\eta c_s}{K}\right)^{0\cdot3} = (6{\cdot}76)^{0\cdot3} \text{ — } \textit{Problem } 3 = 1{\cdot}77$$

$$\frac{K}{d} = \frac{7{\cdot}4 \times 10^{-5}}{9{\cdot}2 \times 10^{-2}} = 8{\cdot}05 \times 10^{-4}$$

$$h_{f2} = 0{\cdot}385 \times (8{\cdot}05 \times 10^{-4}) \times 1{\cdot}77 \times 143$$

$$= 9{\cdot}8 \times 10^{-2} \textit{ B.Th.U./sq. ft./sec./°F.}$$

Then

$$H_a = \frac{1}{1/0{\cdot}115 + 1/0{\cdot}098 + 1/14{\cdot}6} = \frac{1}{8{\cdot}7 + 10{\cdot}2 + 0{\cdot}07}$$

$$= 5{\cdot}3 \times 10^{-2}$$

or 191 *B.Th.U./sq. ft./hr./°F.*

In this problem the fluid in the inner tubes has entered at 90° C. and left at 50°, while the shell fluid, flowing counter-current, enters at 20° and leaves at 60°. The temperature difference at both ends of the exchanger is the same, but this is not always the case. In general, the temperature difference causing heat flow will vary, and will diminish steadily with time allowed for interchange.

Consider a typical heat interchanger of surface S in which the temperature difference varies from θ_1 to θ_2. Let the total heat capacity of the two fluid streams passing in unit time be Q. At any small unit of surface ds, the heat flow dH will be given by the equation

$$dH = H_a \theta dS$$

Then the rate of decrease of temperature difference is given by the general relation

$$-d\theta = \frac{dH}{CQ} \text{ or } dH = -CQd\theta$$

where C is some constant factor determined by conditions of flow. It follows that

$$H_a \theta dS = -CQd\theta \text{ or } -CQ\frac{d\theta}{\theta} = H_a dS$$

This equation may be integrated between the limits θ_1, θ_2 and 0, S to give

$$CQ \log_e \frac{\theta_1}{\theta_2} = H_a S$$

The total heat flow $H = CQ(\theta_1 - \theta_2)$. If a mean effective temperature difference θ_m be defined, so that $H = H_a S \theta_m$, then substituting for $(H_a S)$ and for H

$$CQ(\theta_1 - \theta_2) = CQ \log_e \frac{\theta_1}{\theta_2} \theta_m$$

and finally

$$\theta_m = \frac{\theta_1 - \theta_2}{\log_e \theta_1/\theta_2}$$

This is the same logarithmic mean which was found to apply with a regular change in surface, and should always be used in heat-flow problems when the temperature difference is changing with time at a rate proportional to its own magnitude.

If, in a tube and shell heat exchanger, the fluid in the tubes is making more than one pass, the simple relation between inlet and outlet temperature differences no longer applies. A value for the effective temperature difference is not easy to obtain, and some complicated formulae have been suggested.* However, design on the basis of a normal logarithmic mean difference gives a reasonable

* E.g. Gardner, *Ind. Eng. Chem.*, 1941, 33, 1215, and 1495.

approach to the problem, and these cases will not be further considered.

It is frequently the case that large changes in viscosity occur in heat interchange, and not only the temperature difference but the respective film coefficients and hence the overall coefficients may alter very considerably. It is not possible to propose any mathematical solution for such cases which would be simple enough to be of practical use. An approximate formula has been proposed in cases where the value of the overall coefficient varies with temperature difference in a manner that may be taken as linear. In this case the amount of heat transferred per unit area is given by the formula

$$\text{Heat} = \frac{H_{a2}\theta_1 - H_{a1}\theta_2}{\log_e H_{a2}\theta_1/H_{a1}\theta_2}$$

Here H_{a1} and θ_1 are the coefficients and temperature differences at one end of the exchanger and H_{a2} and θ_2 the corresponding values at the other.

When the flow of heat involves a change of state, as in condensation or in boiling, conditions become much more complicated. It is possible to analyse the case of vapour condensation on a surface if it is assumed that the condensation takes place as an even film of fluid covering the solid. In practice it may be shown that this filmwise condensation, although fairly general, can be replaced by a dropwise system in which the condensed liquid gathers into drops, leaving large areas of the surface exposed to the vapour. As would be expected, this dropwise fashion gives much larger coefficients of heat transfer than the film, to which the drops tend to revert on prolonged working. It is reasonably safe to calculate on the basis of filmwise condensation, as this will probably be the final state in any piece of chemical plant. For a single pure vapour condensing in normal filmwise fashion on a horizontal tube, the film coefficient is given by

$$\frac{h_f d}{K} = \sqrt[4]{\frac{r\rho^2 d^3 g}{K\eta\Delta T}}$$

—a non-dimensional equation. This expression was originally derived by Nusselt, and is generally known as the Nusselt Equation. It may be more conveniently used in the form

$$h_f = 0{\cdot}725 \sqrt[4]{\frac{r\rho^2 K^3 g}{n\eta d\Delta T}} \text{ B.Th.U./sq. ft./hr./°F.}$$

In this form r = latent heat of condensation in B.Th.U./lb.

ρ = liquid density in lb./cu. ft.

K = liquid conductivity in B.Th.U./sq. ft./hr./° F. per ft.

g = gravitational constant — $4{\cdot}18 \times 10^8$ ft./hr./hr.

η = liquid viscosity in lb./ft. hr.

n = number of tubes vertically above one another.

ΔT = temperature difference in °F. between vapour and wall.

d = tube diameter in feet.

It will be seen that an experimental constant has been incorporated, but agreement with the formula has been rather unsatisfactory for a wide range of published results. Nevertheless, no better correlation has yet been devised.

A similar form for vertical surfaces is given as

$$h_f = 0{\cdot}943 \sqrt[4]{\frac{r\rho^2 K^3 g}{N\eta\Delta T}}$$

where N is the surface height, and results for this have been still more unsatisfactory. This discord may well be due to the presence of small amounts of non-condensable gases in the vapour. These lower the film coefficient to a most marked degree; for instance, Othmer* shows that the presence of about 0·5% of air in steam lowers the film coefficient by 50%. A further cause for dubiety arises from the temperatures which are to be assumed for the liquid film. This film may have a temperature anywhere between that of the condensing vapour and the tube wall, a range that may be wide. As a result the viscosity of the film may vary widely, and it may not be possible to ascribe a mean value to this. In these circumstances it is generally necessary to apply empirical corrections, or to take a simple overall heat transfer coefficient based on experience.†

Example 5. *Determine the film coefficient of heat transfer from condensing steam at 5 lb./sq. in. gauge across a thin copper wall to water boiling at atmospheric pressure.*

The saturation temperature of the steam is 227° F., and the film of condensate will be at about 212°, the wall temperature. Then the liquid viscosity is $6{\cdot}8 \times 10^{-1}$ *lb./ft. hr., density 59·7 lb./cu. ft., conductivity 0·415 B.Th.U./sq. ft./hr./° F. per ft., and* $r = 970$ *B.Th.U./lb.*

* *Ind. Eng. Chem.*, 1929, 21, 576.

† See *e.g.* Davidson, *Proc. Chem. Eng. Group*, 1948, 30, 56.

$$\text{Then } h_f = 0{\cdot}943\left[\frac{970 \times (59{\cdot}7)^2 \times (0{\cdot}415)^3 \times (4{\cdot}18 \times 10^8)}{3 \times (6{\cdot}8 \times 10^{-1}) \times 15}\right]^{0{\cdot}25}$$

$$= 0{\cdot}943[3{\cdot}39 \times 10^{14}]^{0{\cdot}25}$$

$$= 4070 \text{ } B.Th.U./sq.\ ft./hr./^\circ F.$$

The case set out above is comparatively simple, as little choice is left concerning the film temperature. Had the water on the other side of the wall been at 60° F., then the properties of the fluid film would have been much less clearly defined. The film temperature in the case of a large temperature drop can be taken as the mean of condensing vapour and wall temperature* as a reasonable approximation. When two immiscible liquids are condensing simultaneously, as in condensers for steam distillation, the joint film coefficient is somewhat higher than would be expected by calculation on the basis of mean values,† a result which has been attributed to a roughening of the film of liquid by droplets of a dispersed phase. The effect of inert gas in the condensing vapour is very marked, as shown by Othmer's results referred to above. It is possible that cases of this kind may be analysed by the use of a constant sensible heat transfer coefficient and a varying condensation coefficient,‡ but no general formula has yet been put forward.

In the case of liquids boiling at a solid surface, the theoretical or dimensional approach fails completely. No general formula has found any acceptance, and all figures for boiling film coefficients are empirical. As a result, data for the rate of heat transfer to boiling liquids are quoted in the form of overall coefficients. It would appear, however, that the following general principles can be deduced for films of boiling liquids:

(*a*) The coefficient increases with temperature difference up to a well-marked maximum and then decreases sharply. This is no doubt due to a " vapour lagging " of the heating surface.

(*b*) Turbulence caused by boiling is so marked that the coefficient is not greatly increased by stirring or mechanical convection, until this becomes so powerful that all vapour bubbles are swept away as soon as they are formed. The boiling film then resolves itself into an ordinary liquid film coefficient. There is considerable doubt in such cases whether any boiling occurs at the hot surface at all,

* Colburn *et al.*, *Trans. Amer. Inst. Chem. Eng.*, 1942, 38, 447.
† Baker and Tsao, *Ind. Eng. Chem.*, 1940, 32, 1115.
‡ Silver, *Trans. Inst. Chem. Eng.*, 1947, 25, 30.

as the pressure gradient required for such high velocities will give a considerable elevation in boiling-point. In the most usual case of this kind—the forced circulation evaporator—boiling occurs as flash evaporation at the end of the heating tubes.

(*c*) Surface conditions existing at the interface between liquid and heating surface can alter the actual formation of vapour bubbles and hence the rate of heat flow. It has been claimed that the addition of a surface tension reducing agent can increase the coefficient of heat transfer, but full experimental confirmation appears to be lacking.

(*d*) In ideal conditions for heat transfer it appears that values for the boiling film coefficient approximate to those for the condensing film of a similar liquid. Thus for steam-heated evaporators overall coefficients up to 1000 B.Th.U. sq. ft. hr. ° F. have been obtained. For steam side coefficients of the order of 4000, and a wall coefficient of 2000 (0·1 in. steel), this would indicate a boiling film coefficient of 1000. The boiling film is, however, much more sensitive to conditions than the condensing vapour film. Cichelli and Bonilla* noted that it was impossible to obtain reproducible results with the simplest surfaces unless they were kept absolutely clean. The same workers showed how rates varied with pressure, reaching a maximum possible value with a pressure range approximating to one-third of the critical pressure for the liquid being tested.

(*e*) As in other cases of heat transfer, water and dilute aqueous solutions represent ideal substances for heat flow in boiling, and other liquids give much lower coefficients.†

Example 6. *Coal-tar vapours at 250° C. are condensing round vertical tubes, 3 ft. in length, arranged to boil coal tar at a temperature of 150° C. The tubes are of steel, 4 in. internal diam. × 4·4 in. outside diam.*

Suggest a value for the overall heat transfer coefficient.

Applying Nusselt's Equation to the condensing film, it may be assumed that

$$r = 150 \text{ B.Th.U./lb.}$$

$$\rho = 60 \text{ lb./cu. ft.}$$

$$K = 7 \times 10^{-2} \text{ B.Th.U./sq. ft./hr./° F. per ft.}$$

$$\eta = 7{\cdot}5 \text{ lb./ft. hr.}$$

$$\Delta T = 180° \text{ F.}$$

* *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 755.

† See *e.g.* Rumford, *J.S.C.I.*, 1947, 66, 309.

Then the condensing film coefficient will be

$$0{\cdot}943\left[\frac{150 \times (60)^2 \times (7 \times 10^{-2})^3 \times 4{\cdot}18 \times 10^8}{3 \times 7{\cdot}5 \times 180}\right]^{0{\cdot}25}$$

$$= 41 \ B.Th.U./sq.\ ft./hr./^\circ F.$$

The boiling film coefficient may be assumed to have the same value. The wall coefficient, with K for steel = 26, will be 1560 *B.Th.U./sq. ft./hr./° F.*

The value for $H_a = \dfrac{1}{1/41 + 1/41 + 1/1560} = 20$ *B.Th.U./sq. ft./hr./°F.*

It has already been stated that very few viscous flow heat transfer coefficients are of practical importance. There is, however, one case of natural convection, generally causing viscous flow, which is of sufficient importance to be considered in an elementary work such as this. The rate of heat loss by convection from an exposed surface in still air is of first-rate industrial importance, as leading to poor thermal economy in all types of chemical plant. This rate will vary with the shape and position of the hot surface, but may be summarised into the standard equation.

$$\text{Heat loss} = C \times 0{\cdot}3 \times \theta^{1{\cdot}25} \text{ B.Th.U./sq. ft./hr.}$$

where θ is the temperature difference between the surface and the surroundings in °F., and C has the following values:

(1) Plane vertical surface	1
(2) Plane hot horizontal surface, facing up	1·3
(3) Plane cold horizontal surface, facing up	−0·65
(4) Values (2) and (3) facing down—reversed values	
(5) Horizontal cylinder, diam. 1 cm.	2·0
,, ,, 10 cm.	1·3
,, ,, 100 cm.	1·2
(6) Large irregular surfaces	1·0

Radiation in Heat Transfer. The whole mechanism of heat flow by radiation is much more complicated than in the simpler processes dealt with above. Heat flow is no longer proportional to temperature difference directly, but to the difference between the fourth powers of the absolute temperatures of radiating and absorbing surfaces. The character or emissivity of the surface also determines the rate at which heat may be radiated or absorbed. For

heat interchange between two black surfaces (emissivity e = unity) the rate of heat flow is

$$0{\cdot}173\left[\left(\frac{T_1}{100}\right)^4-\left(\frac{T_2}{100}\right)^4\right] \text{ B.Th.U./sq. ft./hr.}$$

where T_1 and T_2 are temperatures of the surface in °F. absolute. When the emissivities of the respective surfaces are e_1, e_2, then the heat flow becomes

$$0{\cdot}173\, e_1e_2\left[\left(\frac{T_1}{100}\right)^4-\left(\frac{T_2}{100}\right)^4\right] \text{ B.Th.U./sq. ft./hr.}$$

In many industrial plants a hot surface is losing heat to cold surroundings of irregular form, and the emissivity of the hot radiating surface is the only important factor, the emissivity of the surroundings being taken as unity.

Example 7. *Calculate the rate of heat loss from a vertical wall, 10 ft. × 10 ft., at a mean temperature of 100° C., with surroundings at 15° C. The emissivity of the wall surface may be taken as* 0·9.

Heat loss will be by radiation and natural convection.

The radiation loss will be

$$0{\cdot}173\times 0{\cdot}9\left[\left(\frac{212+460}{100}\right)^4-\left(\frac{60+460}{100}\right)^4\right]\times 100$$

$$= 20{,}450 \textit{ B.Th.U./hr.}$$

The convection loss, from the previous section, will be

$$0{\cdot}3\times(212-60)^{1{\cdot}25}\times 100 = 16{,}000 \textit{ B.Th.U./hr.}$$

Total heat loss from wall $= 36{,}450$ *B.Th.U./hr.*

Heat transfer in furnaces, where large volumes of hot gas are involved, is more complicated. The radiation from a solid surface is so much more effective than gas radiation that in small industrial units the gas effects may be neglected, and calculations based on the equations above. The effective surface for giving or receiving radiation will be measured by the solid angle subtended by the active partner in the transfer. This calls for complicated correction formulae, too extensive to be considered here; in the case of two plane surfaces of limited extent, separated by a gap of the same order of dimension, a correction factor of 0·5 may be assumed.

Example 8. *A muffle type furnace, heated from below, has a floor area 20 ft. × 10 ft., and the receiving surface is a bank of steel tubes, staggered to cover the entire ceiling of the muffle, of similar area to the base and at a height of 6 ft. The furnace floor is at 2000° F. and the pipes at 600° F., and emissivity of both may be taken as 0·9. What will be the rate of heat transfer by radiation?*

The radiation exchange per sq. ft. will be

$$0{\cdot}5 \times 0{\cdot}173 \times 0{\cdot}9 \times 0{\cdot}9\left[\left(\frac{2460}{100}\right)^4 - \left(\frac{1060}{100}\right)^4\right]$$

$$= 2{\cdot}5 \times 10^4 \ B.Th.U./sq.\ ft./hr.$$

Total heat flow $= 5{\cdot}0 \times 10^6$ *B.Th.U./hr.*

If the radiating agent is a current of hot gas, the heating effects become still more involved. The only effective radiators among the commoner gases are carbon dioxide and water vapour, while the radiation from these is not a simple function of the absolute temperature. As might be expected the depth of the radiating gas stream also affects the amount of heat transmitted. Thus the total radiated heat from a gas stream of effective depth s and having a molar fraction p of $(CO_2 + H_2O)$ will be $s \times p \times f(T)$, where $f(T)$ is a function of the absolute gas temperature.

Typical values of s, the effective gas stream depth, are shown below:

Form of Gas Stream	Corresponding Value of s
Cylinder of diameter D	D
Space of depth D between parallel planes of infinite extent.	$1{\cdot}8\ D$
Bank of tubes of diameter D, having staggered pitch of clearance D.	$2{\cdot}8\ D$
Rectangular conduit—smallest dimension D .	$1{\cdot}3\ D$

For the evaluation of $f(T)$ it is necessary to rely upon empirical charts. The most generally accepted figures are those given by Schack* and values based on these are set out in Figs. II-5 and 6. In using such charts, the emissivity and temperature of a receiving surface can be allowed for by assuming that this surface radiates as another gas stream. Then the heat gained from a gas stream by a cold body will be

$$e \times (p \times s) \times [f(T_1) - f(T_2)]$$

* "Der Industrielle Warmeubergang".
See also "Technical Data on Fuel." Edit. by H. M. Spiers, 1942.

where T_1 is the temperature of the gas stream and T_2 that of the receiving body.

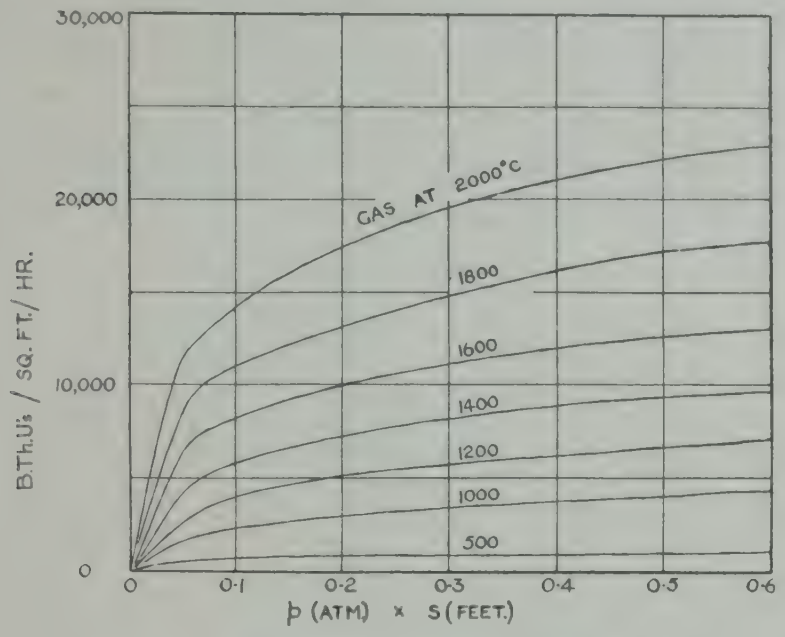


Fig. II–5.—Radiation from Carbon Dioxide.

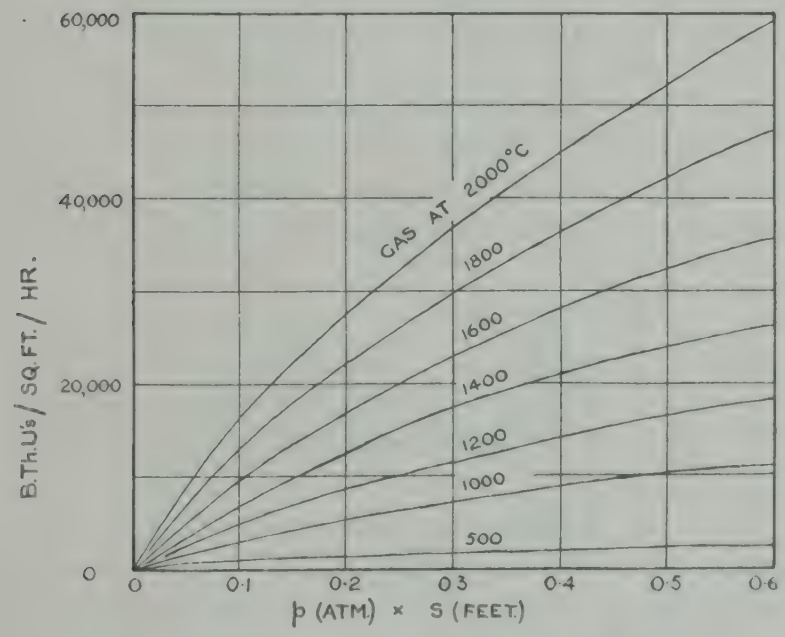


Fig. II–6.—Radiation from Water Vapour.

Example 9. *A waste gas stream at 2000 C., containing 10% of CO_2 (dry basis) and 15% of water vapour, is sweeping over a bank of 3-in. diam. tubes in an oil still. The tubes are set on 6-in. centres*

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and the mean tube wall temperature is 250° *C., with an emissivity of* 0·9. *Calculate the radiant heat transmission to the tubes from the gas.*

The CO_2 *is* 10% *on* 85% *of the total gas, or* 8·5% *of the whole. The shape factor s is* 2·8 × 3 *in., or* 0·7 *ft.*

Then for H_2O $(s \times p) = 0{\cdot}15 \times 0{\cdot}7 = 0{\cdot}1050.$

$$CO_2 \ (s \times p) = 0{\cdot}085 \times 0{\cdot}7 = 0{\cdot}0595.$$

From Figs. 5 *and* 6, *heat radiated at* 2000° *C. will be*

H_2O—16,500 *B.Th.U./sq. ft./hr.*

CO_2—11,500 „ „ „

and at 250° *C.*

H_2O—750 *B.Th.U./sq. ft./hr.*

CO_2—350.

The total heat radiated will be

$$0{\cdot}9\ (28{,}000 - 1100) = 24{,}200\ B.Th.U./sq.\ ft./hr.$$

These two calculations of heat flow by radiation have been made much simpler than the usual case met with in practice. Where the hot gas contains solid particles, it becomes luminous, and the radiation is greatly increased. The radiation effects of CO_2 and H_2O are not quite independent, as each tends to blanket the other to some extent, while the simultaneous effect of hot walls and radiating gas streams may need to be considered. When such cases are encountered, reference should be made to a more detailed book on the subject.*

Pressure Drop in Heat Transfer. It is clear from the preceding pages that high fluid velocities are generally desirable in all heat-transfer equipment. Where high rates of heat flow are to be combined with compactness, special convective heat exchangers, such as that shown in Fig. II–7, may be constructed. This heat exchanger has spiral channels of small depth on either side of thin metal plates; hot and cold liquid flow through these at very high velocities, which automatically involve a considerable pressure drop. The maximum velocity, and hence the highest rate of heat transfer, will be determined by the permissible pressure drop. The size of a heat-transfer unit for a given load is determined by an

* E.g. Trinks, "Furnace Design"; Stoever, "Industrial Heat Transmission"; Fishenden and Saunders, "Heat Transfer."

economic balance between cost of plant and cost of power for pumping. The higher the pressure drop the higher the heat-transfer coefficient, and hence the smaller the plant. The power requirements for pumping rise in the opposite direction. There is often another economic choice to be made between plant size and quantity of cooling water required, which again may increase pumping costs while decreasing plant size owing to increase temperature drop.

The most important contributing causes to the overall pressure

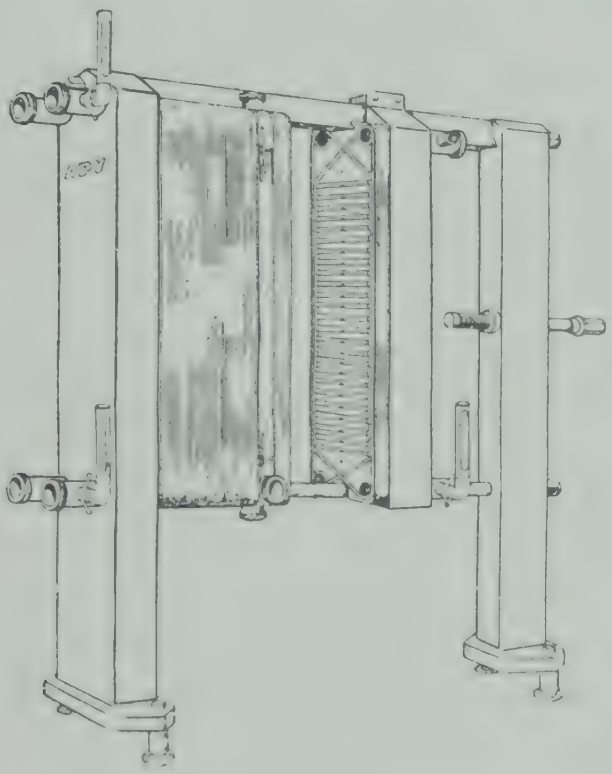


FIG. II–7.—A.P.V. HEAT EXCHANGER.
(Courtesy of Aluminium Plant and Vessel Co. Ltd.)

drop in the flow of a liquid or gas through a heat-exchanging system are:

(1) Frictional loss due to flow through the various conduits.
(2) Losses due to changes in direction of fluid.
(3) Losses caused by velocity changes in the fluid stream.

The first of these is much the most important, and has already been dealt with in the chapter on Pumping. The standard equation for pressure drop by fluid friction will apply—

$$P \text{ (lb./sq. ft.)} = 8\left(\frac{R}{\rho v^2}\right) \cdot \frac{L}{d} \cdot \frac{\rho v^2}{2g}$$

In a heat exchanger, which may be of intricate design, a large number of changes of direction and of velocity will usually occur. These cause a loss of pressure which is normally expressed in terms of "velocity heads" or multiples of the value of $\rho v^2/2g$ for the fluid stream. Thus, for a reversal of flow, or change of direction through 180°, as occurs in the header of a multi-pass tube and shell exchanger, the loss will be from one-half to one "velocity head" in lb./sq. ft.

When an abrupt change in velocity from v_1 to v_2 occurs, the pressure drop is given by the change in velocity head plus a further correcting factor, embodied in the general expression

$$P \text{ (lb./sq. ft.)} = \left[\frac{v_2^2 - v_1^2}{2g} + K\frac{v_2^2}{2g}\right] \times \rho$$

in which K varies with the ratio v_2/v_1 as follows:

v_2/v_1	2·0	3·0	4·0	8·0	10·0
K	0·19	0·27	0·32	0·41	0·43

Some of this lost pressure may be regained when the passage is enlarged again, with a consequent drop in velocity, but it is better to ignore this possibility in a calculation to allow for other possible losses.

Example 10. *A tube and shell condenser is arranged for four passes of cooling water. The total number of* 0·75*-in. internal diam. pipes is* 160, *each* 4 *ft. long, and the total water rate is* 250 *gal./min. Calculate the pressure drop through the condenser, assuming a mean water temperature of* 75° *F. The ratio of header area to tube area is* 5 *to* 1.

As there are 40 *tubes per pass, the flow path is* 16 *ft. of* 0·75*-in. tube, and each tube takes* 6·25 *gal./min. or* $1{\cdot}67 \times 10^{-2}$ *cu. ft./sec. Then* v *is* 5·5 *ft./sec., while* $\eta = 6{\cdot}1 \times 10^{-4}$ *and* $\rho = 62{\cdot}0$ (*Tables*).

$$\therefore \quad \frac{vd\rho}{\eta} = \frac{5{\cdot}5 \times 6{\cdot}26 \times 10^{-2} \times 62{\cdot}0}{6{\cdot}1 \times 10^{-4}} = 35{,}000$$

From the chart (*Fig. I-3*), *of Chapter I* $(R/\rho v^2) = 0{\cdot}0033$.

$$\textit{Then} \quad P = 8 \times 0{\cdot}0033 \times \frac{16}{6{\cdot}26 \times 10^{-2}} \times \frac{62 \times 5{\cdot}5^2}{64{\cdot}4}$$

$$= 196 \textit{ lb./sq. ft.}$$

There are three reversals of flow in a four-pass exchanger, for each of which the pressure drop will be at least

$$0{\cdot}5 \times \left(\frac{62 \times 5{\cdot}5^2}{64{\cdot}4}\right) \text{ or } 15 \text{ lb./sq. ft.}$$

The entry loss on passing from header passage to tubes $(v_2/v_1 = 5{\cdot}5/1{\cdot}1)$ *will be*

$$P = \left(\frac{5{\cdot}5^2 - 1{\cdot}1^2}{64{\cdot}4} + 0{\cdot}34\,\frac{5{\cdot}5^2}{64{\cdot}4}\right) \times 62{\cdot}0 = 38 \text{ lb./sq. ft.}$$

This pressure loss is incurred four times.

The total pressure drop will be

$196 + (3 \times 15) + (4 \times 38) = 393$ *lb./sq. ft.* (*or* $2{\cdot}75$ *lb./sq. in.*)

This section includes approximate methods for the calculation of all the main processes of heat transfer. No attempt has been made to deal with the various methods of electrical heating, as these are usually too expensive for chemical plant work. They may be applied in certain special cases, where heat is required at one particular point, as in dielectric heating, or for certain special types of radiation. Again, no reference is made to the special heat-transfer fluids which have been devised for convective heat flow at high temperatures. For information on these it is necessary to refer to the original publications.

CHAPTER III

DISTILLATION

The separation of two or more components of a mixture by distillation, when all the materials concerned have a definite vapour pressure, has been one of the most widely studied processes of chemical engineering. The necessity for a systematic investigation of distillation conditions first arose in the alcohol industries, followed by those of coal tar and petroleum. These three, and particularly the last, utilise distillation on an enormous scale, and most standard equipment has been evolved with one or the other of these processes in mind.

The general problem of distillation may be defined in the following terms. A mixture of N components, a, b, c . . ., is to be separated into a product P and a residue W, while the amount of certain components in both product and residue must not exceed a certain arbitrary figure.

If the original mixture (F) and the end mixtures (P, W) are expressed in mols, then $F = P + W$.

Also, if the concentration, or molar fraction, of any component a be x_{af}, x_{ap}, x_{aw}, in feed, product, and residue respectively, then

$$Fx_{af} = Px_{ap} + Wx_{aw}$$

The relation between vapour pressure and temperature, even for a single pure substance, is complicated. It can, however, usually be expressed in the form of a linear relation $\log p = A - \frac{B}{T}$, where A and B are constants and T is the absolute temperature. From this can be derived the more convenient form of relation known as Duhring's Rule, which states that if the temperatures at which two similar liquids exert the same vapour pressure are plotted against each other, a straight-line relation is obtained. Then if a suitable reference compound has known vapour-pressure relationships, and two points on the vapour-pressure–temperature curve of a second

compound are known, the vapour pressure of this second substance may be determined throughout the known range of the reference compound. In Fig. III–1, *n*-heptane has been chosen as the reference, and values for the vapour pressure shown along the *X* axis, together with the regular temperature scale. Along the *Y* axis is another regular temperature scale for the "unknown"

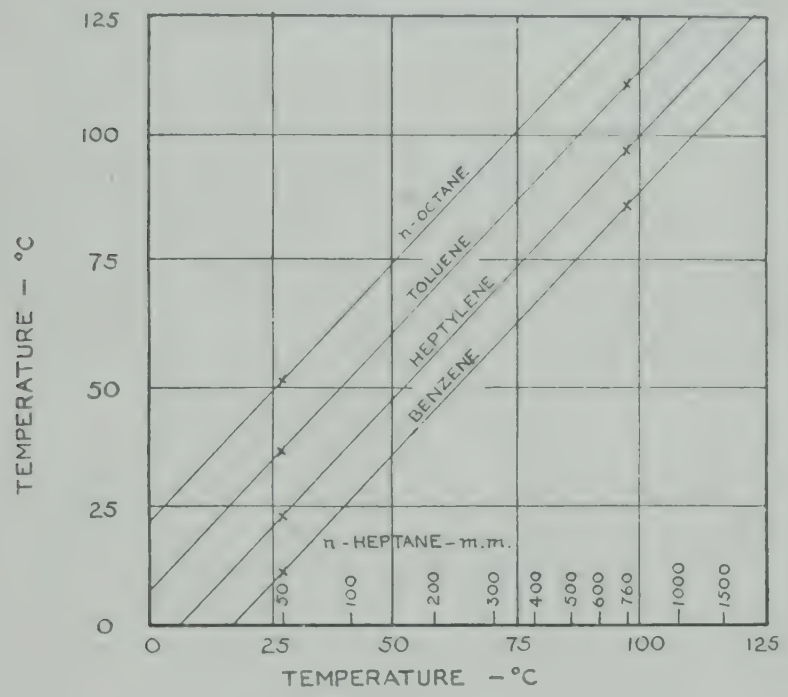


Fig. III–1.—Duhring Relationships.

compounds *n*-octane, benzene, toluene, and a heptylene of b.pt. 95° C.

From published data the following vapour-pressure–temperature relations are known:

Compound	Temperature for a Vapour Pressure of 50 mm. Hg (°C.)	760 mm. Hg (°C).
Benzene . . .	12·2	80·1
Toluene . . .	36·3	110·6
n-Octane . . .	50·6	125·6
Heptylene . . .	?	95·0
n-Heptane (reference) .	27·2	98·4

Points for the first three compounds are shown as ordinates on the *Y* axis of Fig. III–1, against the reference material on the *X* axis. The three lines obtained by joining points for each substance are near enough to parallelism to allow the fourth line, for heptylene,

to be drawn down from the fixed boiling-point temperature. The chart can now be used to read off vapour pressures for the four "unknown" substances. For example, benzene has a vapour pressure of 500 mm. at 67·5°, heptylene the same at 81°, toluene at 96·5°, and octane at 111° C. These agree with published figures to within 0·5° C., which is quite close enough for most calculations.

The relation between vapour pressure and temperature becomes much more complex when mixtures are involved. In an ideal homogeneous solution, the partial vapour pressure of any component at a given temperature is proportional to the molar fraction of the pure component in the mixture.

Then $P_a = Ax_a$, where A is a proportionality constant. Consideration of the limiting value when $x_a = 1{\cdot}00$ will show that A must be the vapour pressure of the pure component. Mixtures which show this relationship can be defined as obeying Raoult's Law. Non-ideal solutions which do not follow this law may be correlated by introducing a correction factor (λ_a) so that $P_a = \lambda_a A x_a$. Then λ_a may be defined as an "activity coefficient", and is either greater or less than unity. In the limiting case of a mixture of mutually insoluble compounds, values of λ are greater than unity, the system is defined as showing positive deviations from Raoult's Law, and minimum boiling mixtures are formed. A large number of completely miscible mixtures also show positive deviations, and will form minimum boiling azeotropes when the pure components boil near the same temperature. If the pure components are widely separated in boiling-point, azeotropes will not be formed, but the effect of one substance on the other may be utilised in the so-called "extractive" distillation, a process to be dealt with later. Conversely, when two compounds have an affinity for each other, the activity coefficients are less than unity and the system is said to show negative deviations from Raoult's Law. Maximum boiling azeotropes can be formed, or alternatively the boiling-point of one component of a mixture can be selectively raised. The values of λ for any component of a mixture may vary widely with temperature or concentration, but the first mixtures to be studied here will be assumed to follow Raoult's Law, when λ is constant at 1·00.

Using this law it is possible to calculate, for any mixture of known liquid composition, the boiling-point and composition of the equilibrium vapour. An example of this can be based on the five hydrocarbons featured in the graph of Fig. III–1.

Example 1. *A mixture of n-heptane (C_7H_{16}), heptylene (C_7H_{14}), octane (C_8H_{18}), benzene (C_6H_6), and toluene (C_7H_8), in equal parts by weight, is raised to the boiling-point. Calculate the initial vapour composition and boiling temperature at 760 mm. Hg pressure.*

In 100 gm. of the mixture there are

$\frac{20}{100}$ or 0·2	mols heptane		0·1900
$\frac{20}{98}$ or 0·204	,, heptylene	giving, as molar fractions	0·1940
$\frac{20}{114}$ or 0·175	,, octane		0·1665
$\frac{20}{78}$ or 0·256	,, benzene		0·2435
$\frac{20}{92}$ or 0·217	,, toluene		0·2060
1·052 mols			1·0000

The vapour pressures can now be calculated for a trial temperature of 101° C., reading the vapour pressures of the pure substances from Fig. III–1.

	(*a*) *Vapour Pressure of Pure Substance*	(*b*) *Molar Fraction*	*Partial Pressure above Mixture* ($a \times b$)
Heptane	810 *mm.*	0·19	154 *mm.*
Heptylene	850 ,,	0·194	165 ,,
Octane	350 ,,	0·1665	58 ,,
Benzene	1400 ,,	0·2435	340 ,,
Toluene	550 ,,	0·206	113 ,,
Total			830 *mm.*

This is too high a total pressure, so a lower temperature, 99° C., is chosen and the trial repeated.

	(*a*)	(*b*)	*Partial Pressure*	*Molar Fraction*
Heptane	770	0·19	145 *mm.*	0·188
Heptylene	800	0·194	155 ,,	0·201
Octane	320	0·1665	53 ,,	0·068
Benzene	1300	0·2435	316 ,,	0·410
Toluene	500	0·206	103 ,,	0·133
Total			772 *mm.*	1·000

The total pressure is near enough to 760 mm. to warrant the assumption of 99° C. as the boiling-point. Then the molar composition of the vapour is given by dividing the various partial pressures by the total pressure, with the results shown in the last column.

A simple distillation, in a " batch " still, will produce initially a vapour of the composition shown by such calculations as the above. Composition of the liquid, and hence of the vapour, will change steadily as the distillation proceeds. The principal use of these vapour equilibrium calculations is in the design of fractionating columns, in which ideally the vapour evolved on each plate is in equilibrium with the liquid. An exact computation of the changes in composition can only be made by such methods as those shown above, although, as will be seen, a number of approximate methods are available.

Most stills, or boilers for the production of a vapour for further treatment, were originally direct-fired vessels. Perhaps the most important survivor of this type is the pot still of the coal-tar industry. A diagram of this still is shown in Fig. III–2; it is built of riveted steel plates, with an inverted saucer bottom. This last ensures that the pitch residues run off at the relatively cool rim section and not at the bottom centre, which is exposed to the hot gases from the coke fire or producer gas flame. These stills, some 9 ft. 6 in. in diameter by 8 ft. high, take a charge of 15 tons of tar, preheated to 100° C., and distil off about 6 tons of mixed oils in a run of 14 hours, finishing at an end temperature of 300° C. On a basis of 220 sq. ft. of effective heating surface, this corresponds to an evaporation of some 5 lb./sq. ft./hr., an interesting comparison with boiler practice. An old-type Lancashire boiler would evaporate 3·5 lb. of water/sq. ft./hr., but modern practice demands three to four times this rate, with much higher figures for water-tube units. Bearing in mind the relative molecular weights, it will be seen that the heat passing is of the order of ten times the practical rate attained in the pot still. The pot still is therefore not a fast-working unit, nor is it thermally efficient; figures are quoted of the order of 1·5 cwt. of coke per ton of tar used, which suggests an overall efficiency of about 25%. Retention in the industry is due to the fact that a number of small distillations have to be carried out, and the pot still is very elastic in its operation. As a rule no attempt is made to treat the vapours further at this stage, and the various fractions obtained represent equilibrium states between liquid residue and vapour at the various stages of the distillation. The vapours pass through a preheater, in which the next charge for the still is being heated, before going to condensers of cast-iron or wrought-iron pipe coils immersed in tanks of water. This rather primitive construction

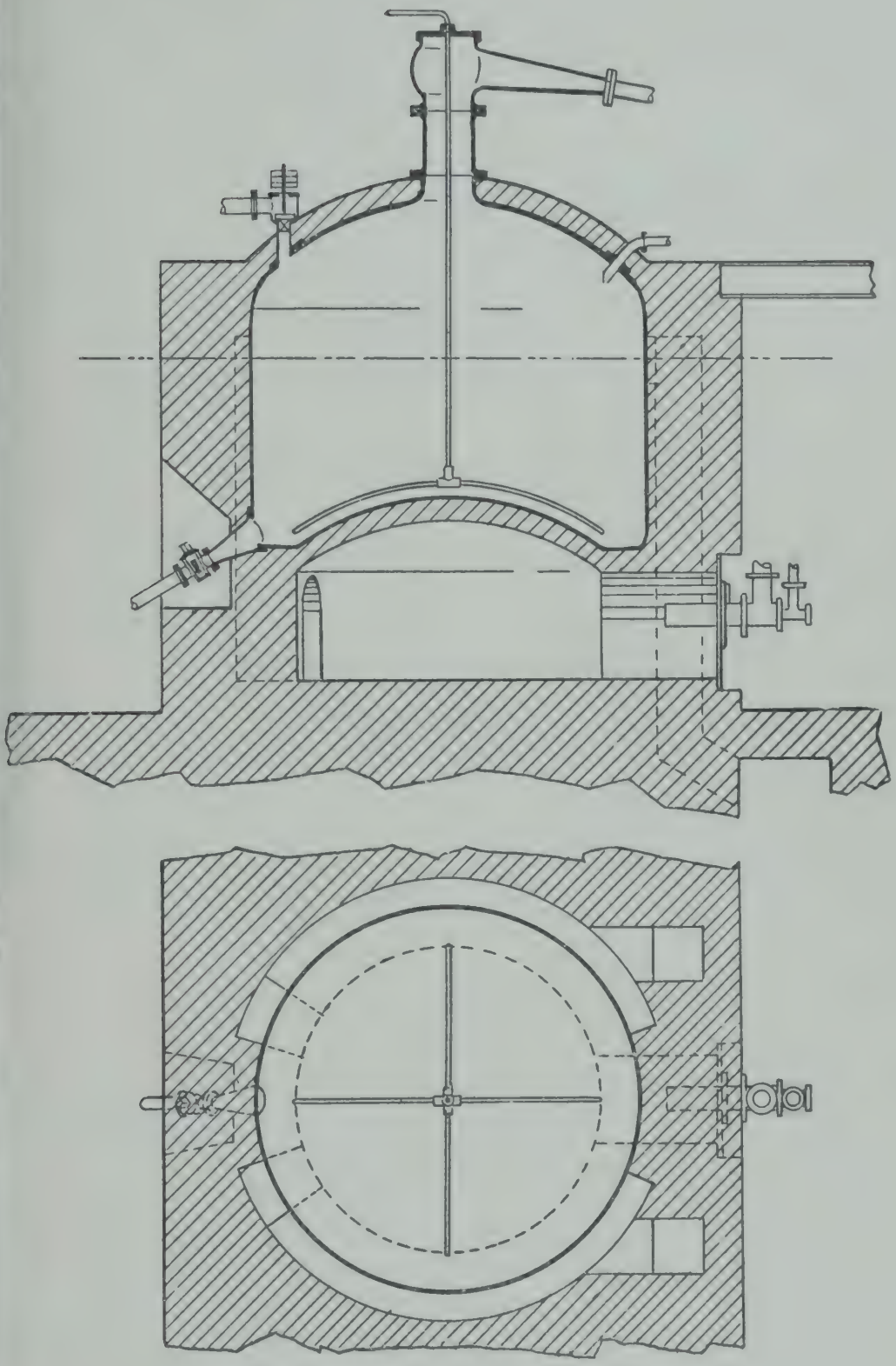


FIG. III-2.—DIRECT-FIRED TAR STILL.

is necessitated by the corrosive nature of the hot tar vapours. The pot stills are too large and thin-walled to work at pressures much above or below atmospheric, and the large " dead-weight " safety valve shown should be arranged to discharge outside the hot brick-work setting. Fire risk with such a large volume of hot inflammable liquid is considerable and is one of the many reasons why these units have been largely replaced by pipe stills.

The conventional pipe still consists of a number of lengths of straight pipe, arranged with a combustion chamber and hot gas flue so that the hot gases flow counter-current to the liquid being

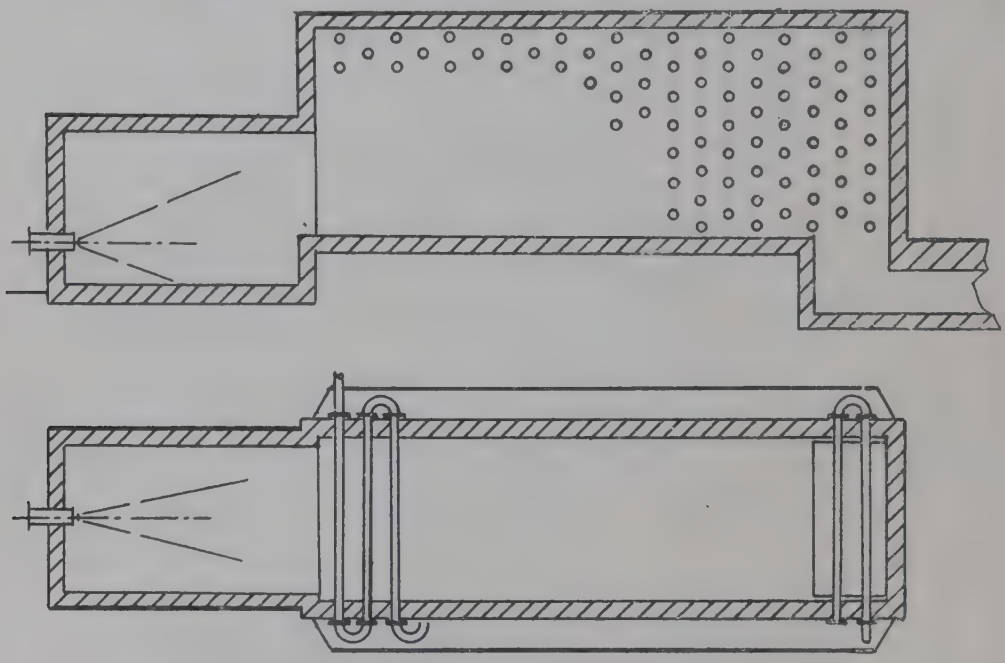


Fig. III–3.—Early Form of Pipe Still.

distilled. The pipe-still principle was first applied in the tar industry, and a diagram of the original layout is shown in Fig. III–3. The pipes are laid in lengths of about 15 ft., with the ends passing through the walls of the hot gas flue. Flanged connecting pieces join up the straight lengths into one continuous channel, some 3000 ft. in length. In this original design the combustion chamber was separated from the actual pipes, but modern designs line the chamber with the pipes, thus making full use of the high rate of heat transfer possible by radiation at combustion temperatures. Arrangements of pipes for this are shown in Fig. III–4; these are all petroleum stills, and it is in this industry that the development of pipe stills from the original form has been carried out.

All pipe stills rely upon pumping the oil to be heated at a high velocity, so that flow is always as turbulent as possible. A minimum value of 10,000 has been suggested for the Reynolds' Criterion, and every precaution must be taken to see that the pumping does not stop. In a typical instance a complete duplicate set of pumps are always kept standing by, and a steam engine can take over the drive if electric power fails. Despite these precautions all pipe stills working to high temperatures choke up slowly with deposited carbon,

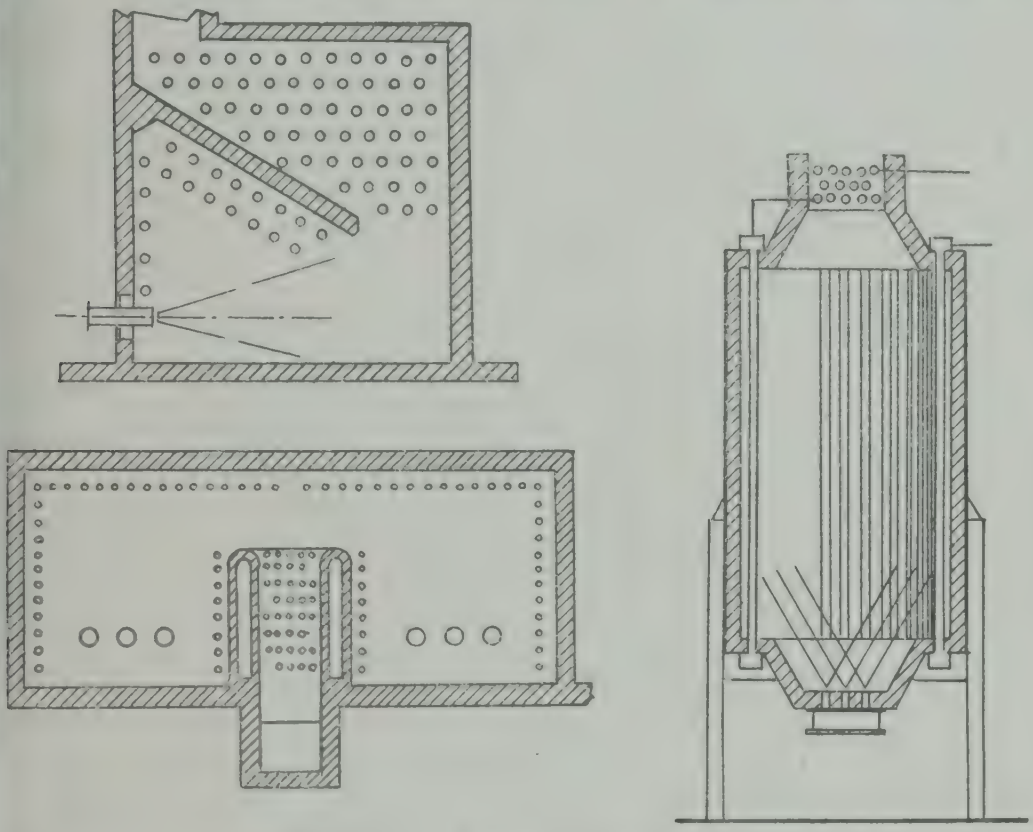


FIG. III–4.—FORMS OF PIPE STILL.

and must then be shut down for cleaning. The flanged sections are removed, without need to break into the actual brickwork of the still heating chamber, and the straight lengths of pipe can be drilled out, or scoured with revolving wire brushes.

The reason for the lack of development of the pipe still in the tar industry was the relatively small quantity of material which was to be treated, except at one or two large works. It is not an economic proposition to build a pipe still for less than about 500 gallons of oil per hour, because of the narrow pipes and the high pressure drop required. This has now been overcome by pipe stills

of comparatively short pipe length and wide bore, in which turbulence is obtained by the recirculation of pitch. A diagram of one of these is shown in Fig. III–5, and it will be seen that the straight pipe has been abandoned in favour of a coil of " figure eight " plan. This suggests that coke deposition has been reduced to a negligible figure, as the pipes could hardly be cleaned by normal methods. They could be burned out slowly at higher temperatures, but the method is attended with considerable risk of damage to the piping. However, such stills have recorded very long uninterrupted periods of operation, and it is probable that the thermal stability of most coal-tar pitch is a contributory factor in this freedom from deposition.

The rates of heat transfer which can be obtained in these stills are limited to some extent by the thermal instability of the oils

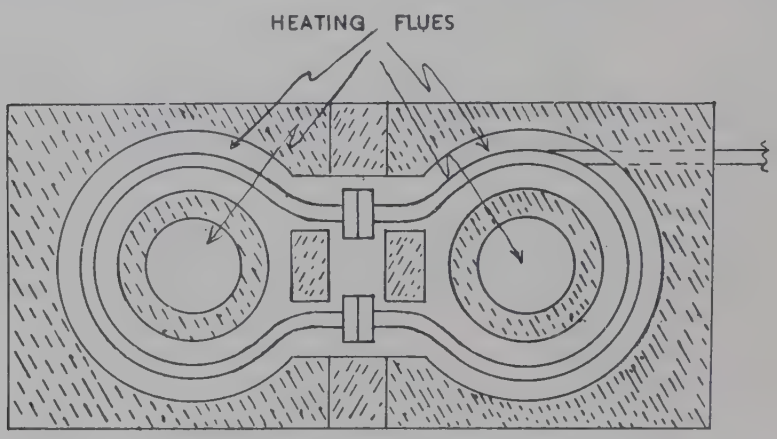


Fig. III–5.—Coiled Pipe Still.

being distilled. It would be possible to obtain actual transfer rates of the order of 25,000 B.Th.U./sq. ft. by radiation alone, as can be seen from the heat-transfer section of this work. It might not be possible for the oil to take up this heat without overheating, followed by thermal cracking at the pipe surface. It is generally recognised that very high rates of heat transfer with liquids can only be obtained by prevention of vaporisation and by high liquid velocities under pressure. The pipe still normally operates under pressure, and all vaporisation occurs when the superheated liquid is discharged into the " flash " chamber. The heat-transfer rate is governed, in the hotter part of the still, by the maximum rate at which the oil can absorb heat without " cracking ", and in the cooler sections by the low coefficients of heat transfer through the gas film. Methods for the calculation of heat flow in both these cases have

already been given in the section on heat transfer. In order to give some idea of the effect of these quantities on pipe-still design, a typical example is given below.

Example 2. *A pipe still is to heat* 2000 *gallons of coal tar from* 100° *to* 400° *C. The pressure in the pipes is sufficient to keep all the tar oils in liquid form. The maximum heat input in the combustion chamber section must not exceed* 10,000 *B.Th.U./sq. ft./hr. The hot gases leave this section at* 1800° *F., and heat transfer in the convection section is at a rate of* 5 *B.Th.U./sq. ft./hr./ F. Suggest a suitable pipe size and length for this duty.*

The mean viscosity (η) *of the tar will be about* 2×10^{-3} *F.P.S. units, and the density* (ρ) 72·5 *lb./cu. ft. Assuming a value of* 20,000 *for the Reynolds' Criterion* ($vd\rho/\eta$) *gives a value for* $v \times d$ *of* 0·552. *But the volume of tar passing per second is* 0·09 *cu. ft., whence the pipe diameter d must be* 0·21 *ft. or* 2·5 *in.*

The heat load, with a specific heat of 0·5, *will be* $6{\cdot}3 \times 10^6$ *B.Th.U./hr. If this is evenly distributed between radiant heat and convection sections, the area in the former will be* $(3{\cdot}15 \times 10^6)/10^4$ *or* 315 *sq. ft.*

In the convection section the tar enters at 212° *F. and leaves at* 482° *F., while the gas enters at* 1,800° *F. and will be assumed to leave at* 500° *F. This gives a mean temperature difference of* 680° *F., and a heat flux of* 3400 *B.Th.U./sq. ft. The area of heat transfer surface in this section will therefore be* 930 *sq. ft.*

A pipe of 2·5 *in. internal diam. will have a mean surface of* 0·68 *ft. per foot run, so that* 460 *linear feet of pipe are required in the radiant heat section and* 1370 *ft. in the convection section.*

It may be noted that if the tar "flashes" to 350° *C. on release, sufficient heat is available to vaporise one-third of the tar (latent heat* 150 *B.Th.U./lb.).*

Calculations of this type are necessarily empirical, and must rely upon test figures. The results given above appear to be in fair agreement with such data. It is possible, by a calculation based upon Raoult's Law, to determine the composition of the vapour which will be flashed off in such a process. This may either be evolved continuously during passage through the pipe, or released as a whole at the outlet when pressure is released, but in either case a vapour is formed which is in equilibrium with the residual liquid.

Suppose that 100 mols of feed contain F_a, F_b, F_c, etc., mols of each component, and that the feed (F) is separated into P mols of

product and W of residue. Let the mols of each component in product vapour be P_a, P_b, P_c . . . and W_a, W_b, W_c . . . in the residue.

If the total pressure of the vapour is Π, then by Raoult's Law $\Pi \times \frac{P_a}{P} = A\frac{W_a}{W}$, where A is the vapour pressure of the pure component. But $P_a = F_a - W_a$, or substituting $\Pi\left(\frac{F_a - W_a}{P}\right) = A\frac{W_a}{W}$ and from this $W_a = \frac{\Pi F_a}{(A \,.\, P/W + \Pi)}$, an expression for the amount of W_a present in the residue.

Example 3. *Suppose the mixture of hydrocarbons given in Example* 1 *is heated in a pipe still to* 101° *C. at atmospheric pressure* ($\Pi = 760$ *mm.*). *What fraction will be vaporised and what will be the composition of the residue?*

Assume a trial value of P/W *with half the fraction boiling nearest to* 101° *vaporised, along with all lighter components. On this basis,* 45 *mols are left of the original* 100 *and* $P/W = 1{\cdot}22$. *A tabular computation gives the results below.*

	ΣA	ΣF_a	$\Sigma \Pi F_a$	$\Sigma A \,.\, P/W$	$(A \,.\, P/W + \Pi)$	ΣW_a
Heptane . .	810	19·00	14,400	990	1750	8·2
Heptylene . .	850	19·40	14,750	1035	1795	8·2
Octane . .	350	16·65	12,650	427	1187	10·7
Benzene . .	1400	24·35	18,500	1710	2470	7·5
Toluene . .	550	20·60	15,650	670	1330	11·8
						46·7

The residue W *is close enough to the trial value of* 45 *to assume that the values above are correct. A further trial on the basis of* 46 *mols of residue could be made, but differences in composition would be small. Then from each* 100 *mols of feed,* 53·6 *will be vaporised, and the residue,* ΣW_a, *will have the molar composition shown in column* I, *or alternatively the weight composition of column* II.

	I (%)	II (%)
Heptane . .	17·7	18·2
Heptylene . .	17·7	17·9
Octane . . .	23·0	27·0
Benzene . .	16·2	12·9
Toluene . . .	25·4	24·0

The direct-fired stills described above have as wide a range of temperature conditions as can be desired, but when temperatures

are lower, the convenience of steam heating, as well as the avoidance of fire risk, makes it the normal method for boiling the distillation mixture. A typical steam-heated still is shown in Fig. III 6; the heating tubes are so arranged that the whole steam chest may be withdrawn for cleaning from one end of the cylindrical still body. These boiler-form stills may be made up to hold a charge of 20,000 gallons, while 5000-gallon units are commonplace. As a rule the vapours pass to some type of fractionating column, and this should

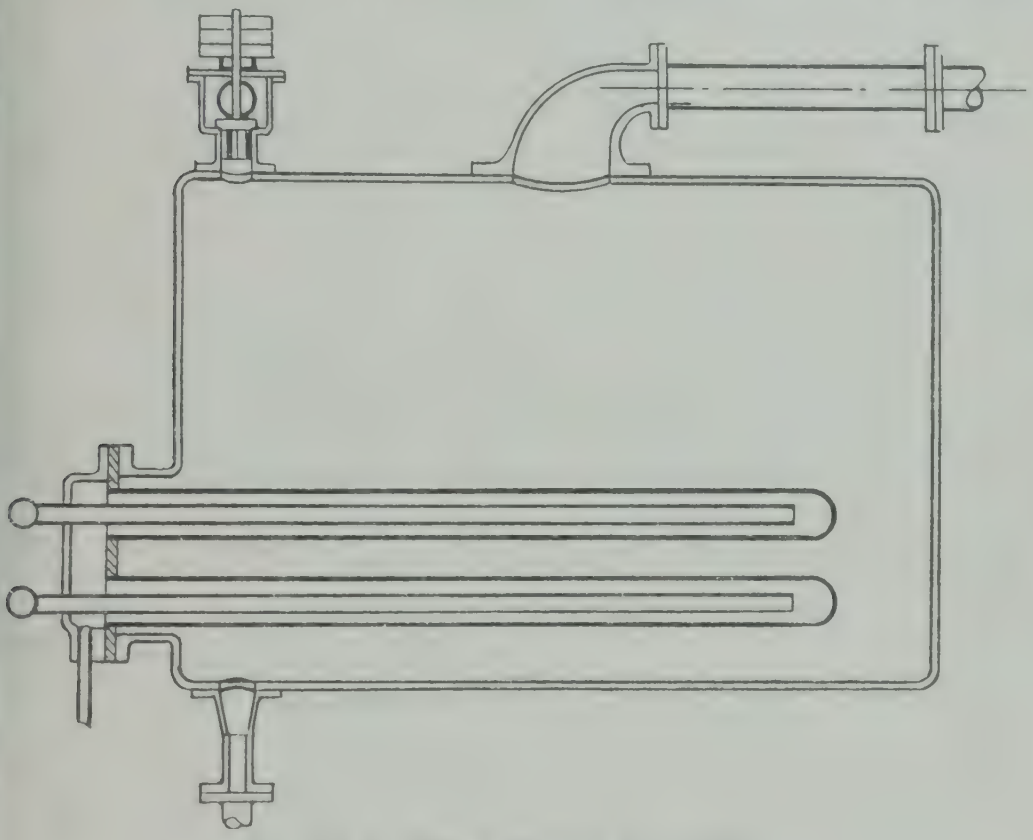


Fig. III–6.—Steam-Heated Batch Still.

never be mounted directly above the still body, but connected by a vapour pipe with a removable section. The column can then be blanked off completely from the still when the latter is to be cleaned; if they are connected during cleaning, there is always the possibility of pockets of heavy vapour flowing down from the column to the still body, and several fatal accidents have been traced to such an occurrence. For stills working under vacuum, special designs which avoid hydrostatic head over heating surfaces have been recommended.*

* Leyland, *Trans. Inst. Chem. Eng.*, 1949.

All these stills make a single " cut " between vapour and liquid, and it is necessary to feed the vapour to some type of fractionating column if any selective distillation process is to be carried out. In the fractionating column, continuous interchange between liquid from a condenser and vapour from the still leads to the concentration of lower-boiling constituents of a mixture at the top of the column. The aim of column design is to bring about intimate contact between vapour and liquid, while at the same time keeping both streams flowing evenly in opposite directions, and giving the smallest possible

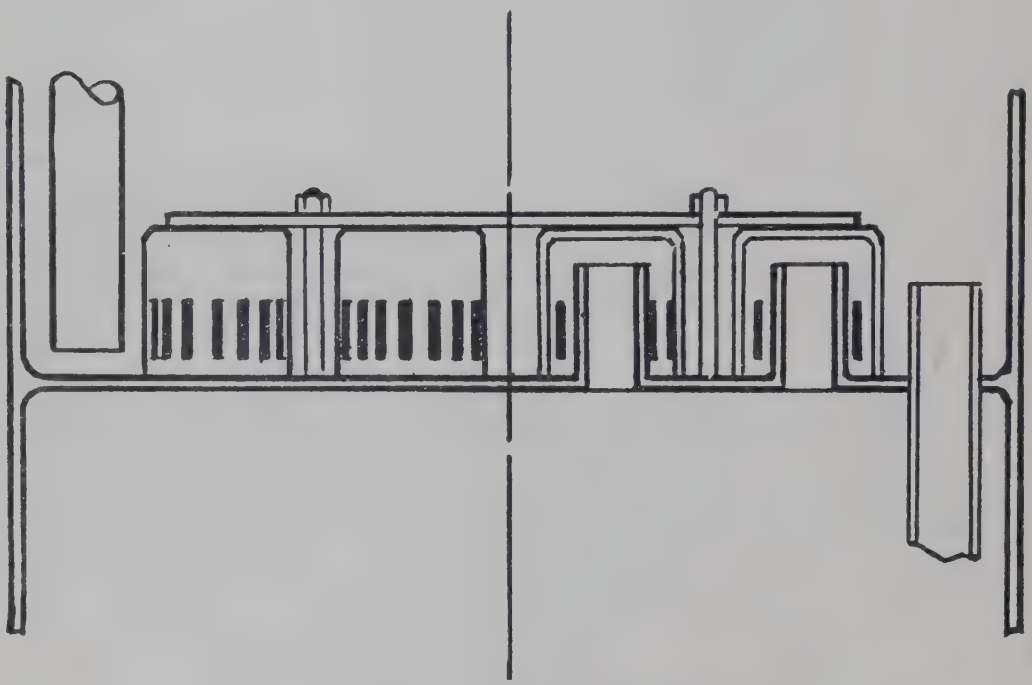


Fig. III–7.—Cap-and-Bubble Plate.

pressure drop. This pressure drop is of vital importance in the design of vacuum distillation plants, but must in any case be considered in plant design. A tall fractionating column containing 30 plates, each giving a back-pressure of 4 in. w.g., means a total additional pressure of about 4·3 lb./sq. in. at the bottom of the column.

The most usual type of fractionating column is built up from " bubble " plates, on which the vapour stream bubbles up through a liquid layer. In the " classic " form of this plate, as shown in Fig. III–7, a number of circular bubble caps, with slits cut in the sides, are set over corresponding vapour uptakes from the space below. A liquid seal, of height fixed by the weir discharge over the

liquid downtake, covers the slits, so that the vapour must bubble through the liquid, which is flowing from side to side of the plate in its passage down the column. This simple arrangement has persisted for a number of years, and many columns are still being built on these lines, but there are a number of faults.

In the first place the fluid head causing liquid flow across the column will give a deeper seal on one side of the plate than on the other; more vapour then passes through the more lightly sealed caps, and in extreme cases liquid may be blown right away from some. Again, the changes in concentration caused by alternating directions of flow are undesirable, as it can be shown theoretically that the highest liquid concentration should always be at the same side of a plate. From a practical standpoint, it has been noted that bubbles formed at the slots often coalesce, decreasing the interface between liquid and vapour, while a single flat plate, in a large-diameter column, is heavy and needs reinforcing if it is not to sag in the middle, increasing the liquid seal on the centre caps. Some attempts to overcome these handicaps are shown in the designs below. In Fig. III—8, showing the " Kuhni " plate, a concentric series of pressed metal annuli, of U section, are arranged alternately upright and inverted. The edges of the inverted U-sections are serrated to break up the vapour stream, the serrations being rectangular, $\frac{1}{2}$ in. long by $\frac{1}{8}$ in. deep. The liquid collects in the middle of each plate, flows down through an inner seal, and out through distribution pipes to the outer edges of the trough layer below. The A.P.V.-West plate (Fig. III–9) relies for the main breaking up of the vapour stream on a perforated plate floor, above which the liquid is held by a weir partition. The liquid downtakes from the weir space are taken across the column between the plates, so that the concentration change is in the same direction on each plate. This form of plate represents an effort to obtain the relatively low pressure drop and good contact which can be obtained by a simple perforated or sieve plate, without the disadvantage of a narrow range of vapour velocities, below which the sieve drains, and above which the column is " liquid-logged ".

All these designs rely upon a positive liquid seal for contact between the two phases. As a result the pressure drop across each plate must be at least that of the seal, and is normally at least twice this figure. The normal seal depth above the top of the slots in the usual bubble cap is about 1 in., but it has been suggested that

smaller seals are practicable in a Kuhni or A.P.V.-West design. Sieve plates appear to be too unreliable for modern use. If a very low pressure drop for a large plate equivalence is necessary, as in vacuum work, then packed columns can be used. These are cylindrical shells packed with various types of filling, either evenly arranged or distributed at random. In normal distillation processes

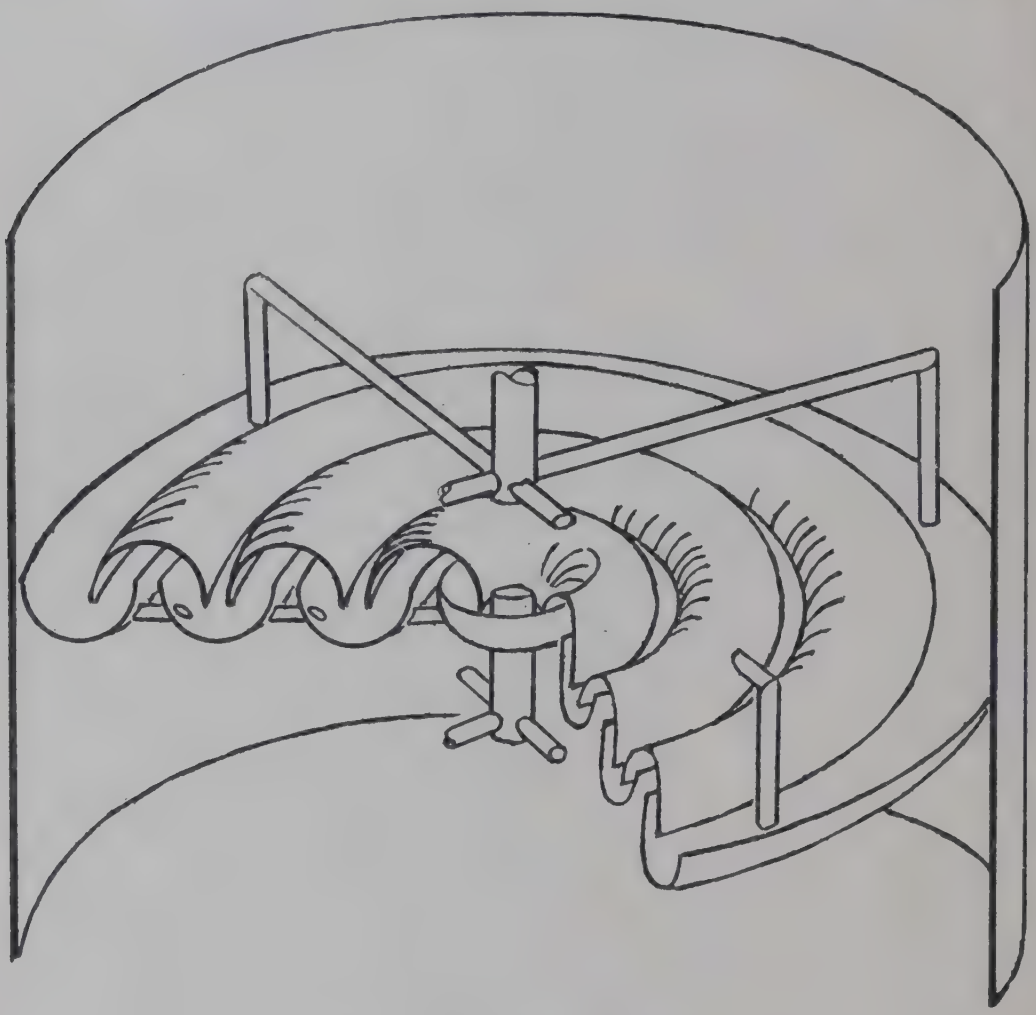


Fig. III–8.—Kuhni Plate.

(*Courtesy of Messrs. Blairs, Govan.*)

the actual ratio of liquid volume to gas volume is low, and in consequence the packing is not evenly wetted, especially in large industrial columns of 3 ft. or more in diameter. The liquid tends to drain to the walls, so that columns of a height equivalent to more than ten bubble plates are unusual. In distillation work the most usual packing is either the Raschig ring, a short cylinder of length

equal to diameter, or the Lessing ring, of the same type but incorporating a central partition. The more complicated forms of packing have been evolved in connection with gas washing, and are dealt with in more detail under that heading. A recent addition to the ring packings is the Lessing ring in wire gauze, but the

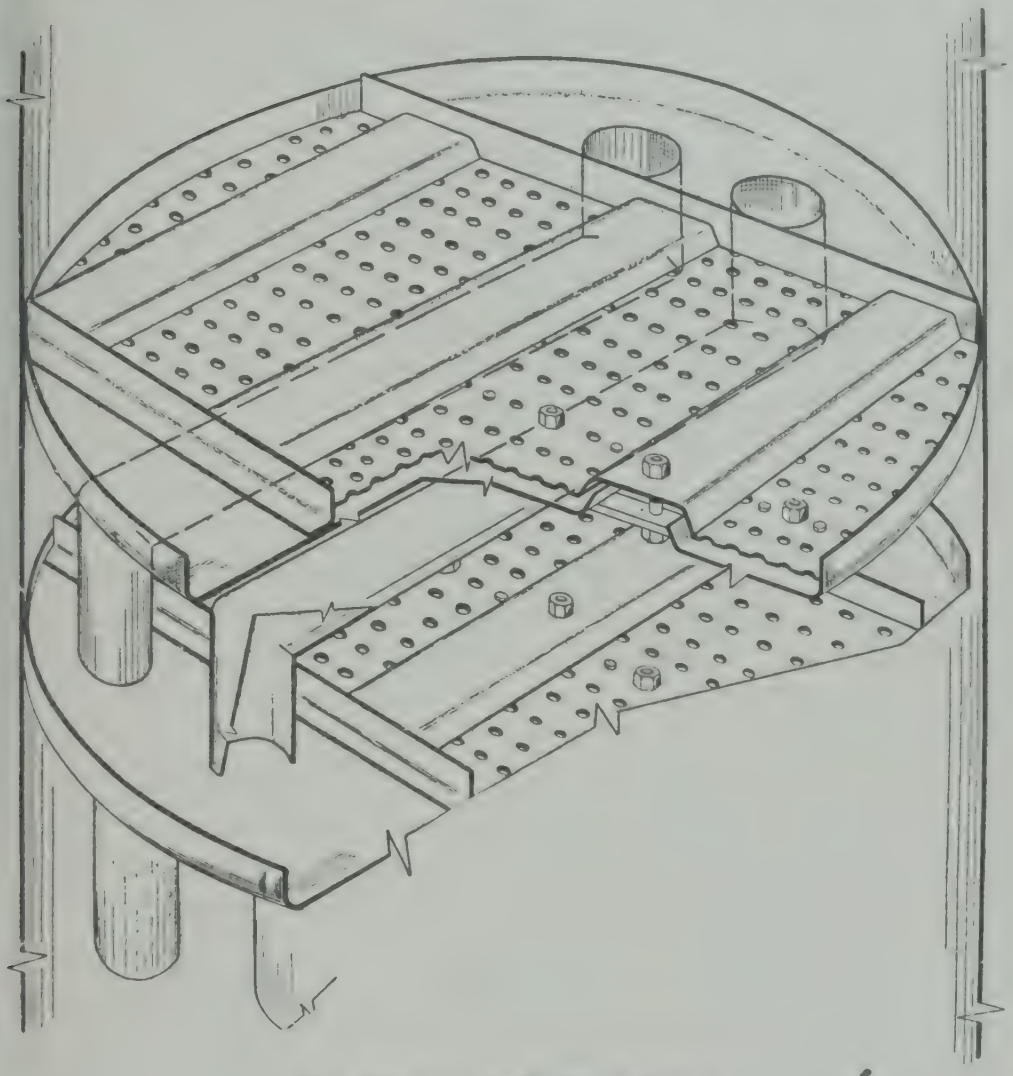


Fig. III-9.—A.P.V.-West Plate.

(*Courtesy Aluminium Plant and Vessel Co.*)

fragility and cost of these will probably make them suitable only for large-scale laboratory work. It is possible that the simplest form of distillation column would be a cylinder, down the walls of which the liquid flows, with vapour passing up the centre; this would give a low pressure drop but poor contact between the phases. Some attempts have recently been made to improve on this by

introducing a central spinning section. Birch and his co-workers,* for instance, describe a column with a central spinning band twisted into a spiral, while others have built units with a rotating central cylinder, leaving a narrow annular ring for mass transfer.† It is doubtful if such columns will find application on anything larger than a laboratory scale, and then for work in the pressure range from 1–10 mm. $Hg_{..}$. Distillation at very low pressures—the so-called "molecular distillation" process—is separately described later on in this chapter.

The capacity of any fractionating column is principally determined by the allowable vapour velocity. When this velocity is too great, the liquid reflux will no longer flow down the column, and the plant is said to "liquid-log". In the case of a bubble-plate column, the maximum vapour velocity gives a back-pressure across the plate equal to the fluid head in the downtake pipe, and at higher vapour velocities the downtake pipes no longer function. With normal bubble caps and a 1-in. seal of water, the pressure thrown per plate varies from 2 in. w.g. at 1 ft./sec. vapour velocity up to 8 in. w.g. at 4 ft./sec. These figures are based on water and steam at 100° C. and atmospheric pressure, with the velocity calculated on the full cross-section of the column. The figures will vary with plate design, liquid seal, and fluid properties, but it may be concluded that the minimum pressure drop for a bubble plate is about 3 mm. Hg. The total area for vapour uptakes is about $\frac{1}{10}$ of the cross-section of the column, and the area of the slots in the caps the same as the vapour uptakes. In a column with closely set plates, even though the critical velocity for liquid-logging is not reached, splashes from the violently agitated liquid may reach the vapour uptakes to the plate above in quantity sufficient to interfere with normal "plate efficiencies"—a term to be dealt with later. For this reason, among others, it is customary to allow for a plate spacing in the column equal numerically to the vapour velocity (i.e. a vapour velocity of 2 ft./sec. suggests a plate spacing of 2 ft.). This splashing effect is more important than true "entrainment" of small droplets of liquid in the main vapour stream. A general formula for allowable vapour velocity, based on this plate spacing, has been suggested by Souders and Brown.‡ They give the maximum velocity as

* *J.S.C.I.*, 1947, 66, 33.
† Willingham *et al.*, *Ind. Eng. Chem.*, 1947, 39,706.
‡ *Ind. Eng. Chem.*, 1934, 26, 98.

$C\sqrt{[d_2(d_1 - d_2)]}$ lb. vapour/sq. ft. hr. where d_1 = density of liquid in lb./cu. ft., d_2 the density of the vapour in the same units, and C varies from 200 with a plate spacing of 10 in. to 650 with a spacing of 25 in. It follows as a derivation from this formula that d_2v^2 will be almost constant, where v is the allowable linear vapour velocity.

Example 4. *What is the allowable velocity for the vapour in a tar acid column, separating cresols from xylols at a mean pressure of 50 mm. Hg, with a 12-in. plate spacing?*

Ortho-cresol boils at 110° C. at 50 mm. pressure; the vapour will therefore have a density of 0·014 lb./cu. ft. The liquid density will be some 65 lb./cu. ft., and taking for C the value 250 gives the vapour velocity as $250\sqrt{0{\cdot}014}$ *(65) or 238 lb./sq. ft./hr.*

This corresponds to a linear velocity of 4·7 ft./sec. Had the vapour been at atmospheric pressure but the same temperature, the mass velocity would have worked out at 935 lb./sq. ft./hr. and the linear velocity at 1·2 ft./sec.

Vapour velocities in a packed column are of the same magnitude as those in plate columns, but the pressure drop for a column height equivalent to bubble plate in vapour–liquid interchange is about 1·5 mm. Hg, or half that of the bubble system.* Many attempts have been made to reduce this pressure drop, so far without much success; the various forms of "spinning band" column give pressure drops of the order of 0·1 mm. Hg for the same plate equivalence, but are still in the experimental stage.

Liquid flow conditions in a fractionating column are not so critical, and the normal processes of distillation place a limit on the amount of liquid which is flowing down the column. In the case of a plate system, the downtake pipes should be designed to give a liquid velocity of 5–10 ft./min., with a similar velocity over the "weir" at the top for a head of, at most, $\frac{1}{2}$ in. of liquid. It may be necessary to modify plate design in order to achieve these conditions. When packed columns are being used, the balance between liquid and vapour is self-adjusting, as the same channels serve for both, and the liquid flowing down is determined ultimately by the weight of vapour going up. The value of d_2v^2 must be kept constant, as previously stated, so that for pressures of $\frac{1}{10}$ atm. the allowable velocity will be $\sqrt{10}$ times that possible at ordinary pressures. The same rules apply in the design of pipes for joining a still to a column

* See, e.g., Chilton and Colburn, *Ind. Eng. Chem.*, 1931, 23, 913.

or column to condenser. At atmospheric pressure, vapour velocities in these pipes up to 20 ft./sec. may be used; at a pressure of 7·6 mm. Hg velocities up to 200 ft./sec. are possible. This would demand passages of up to ten times the area of those used at normal pressures for conveying the same weight of material. This matter is more fully discussed in a paper by Griffiths,* but fuller consideration is outside the scope of the present book.

The usual arrangement of a distillation system may be represented as in Fig. III–10, where a feed which is either liquid or

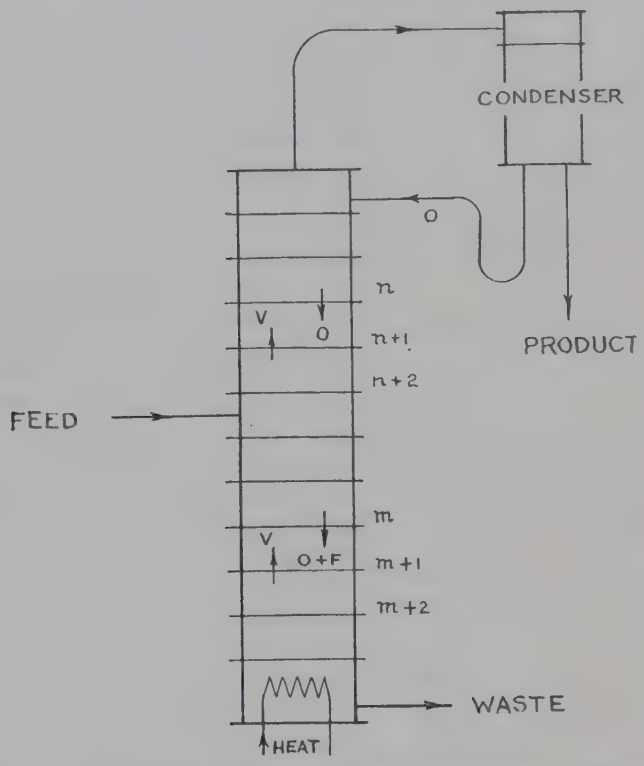


Fig. III–10.—Diagram of Fractionating System.

vapour enters the central section of a fractionating column and comes into contact with a descending stream of liquid and an ascending stream of vapour. At the top of the column the vapour passes to a condenser, from which a portion of the liquid condensate is withdrawn as product, while the remainder returns to the column as reflux. At the base of the column a heating unit partially vaporises the liquid flowing down, with the residue withdrawn as " bottoms " or waste.

In the simple distillation analysis made in this book it will be

* *Trans. Inst. Chem. Eng.*, 1945, 23, 113.

assumed that all components of any mixture have the same molal latent heats, and that these heats are constant throughout the column. This assumption can be made more exact by the use of a modified molecular weight, but is nearly correct for chemically similar substances. Then if there are no heat losses from the column, a state of affairs which can be nearly achieved by efficient lagging, the number of moles of liquid overflow and of vapour is constant from plate to plate, although the relative composition may change, unless some side-stream of material or heat enters the system.

Referring to the diagram (Fig. III 10) at any typical plate above the feed, a mass balance shows that

V (mols vapour) $= O$ (mols liquid reflux) $+ P$ (mols product)

Then for any component A, taking a balance between the n^{th} and the $n + 1^{\text{th}}$ plate from the top,

$$Vy_{a(n+1)} = Ox_{an} + Px_{ap}$$

where y represents the molar fraction in the vapour and x the molar fraction in the liquid, with n, $(n + 1)$ the appropriate plate suffixes. It follows that

$$(O + P)\, y_{a(n+1)} = Ox_{an} + Px_{ap}$$

Dividing through by P, and calling O/P the reflux ratio R,

$$(R + 1)\, y_{a(n+1)} = Rx_{an} + x_{ap}$$

or

$$y_{a(n+1)} = \frac{R}{R+1} x_{an} + \frac{x_{ap}}{R+1}$$

This is a general relation between the vapour rising from a plate and the liquid on the plate above for any *one* component.

In the original statement of the problem of distillation, the composition of the product P was specified as x_{ap}, x_{bp}, etc. As fractionation ceases when the vapour leaves the top plate ($n = 1$), the vapour composition y_{a1}, y_{b1}, etc., will be equal to x_{ap}, etc. The value of $x_{ap}/(R + 1)$, etc., can be calculated for any definite value of the reflux ratio. Reverting now to the feed section of the column, it is clear that the composition of the liquid on the plate to which the feed is pumped will tend to approximate to the feed composition if the feed is liquid. If it is assumed that the feed enters as a liquid at boiling-point, the approximation will be very close, and the feed may be taken as identical with the feed-plate liquid. From the

practical standpoint this arrangement, in which the feed enters as a boiling liquid, is the simplest compatible with thermal economy and is very generally adopted.

In any ideal bubble-plate column the liquid is brought into equilibrium with the vapour by suitable transfer of components between the two phases. If the mixture can be taken to obey Raoult's Law, then a liquid on the feed plate of composition x_{af}, x_{bf}, . . . will give a vapour of composition $P_a x_{af}$, $P_b x_{bf}$ . . ., where P_a, P_b are the vapour pressures of the pure components at some arbitrary temperature T, at which the total vapour pressure is $\Sigma P_a x_{af}$. By trial and error a temperature can be chosen which brings the value of $\Sigma P_a x_{af}$ to a suitable value, such as 760 mm. Hg. The molar fraction of any component A in the vapour mixture will be $P_a x_{af}/\Sigma P_a x_{af}$. Now if the feed plate be designated the $(n + 1)$ plate of the column, these respective molar fractions will be $y_{a(n+1)}$ . . . . The composition of the liquid on the plate above—the n^{th} plate—can now be computed from the formula

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{x_p}{R+1}, \text{ or } x_n = \frac{R+1}{R}y_{n+1} - \frac{x_p}{R}$$

It may be noted that as $\Sigma y_{a(n+1)} = 1 = \Sigma x_{ap}$, then Σx_{an} must also be equal to unity, which forms a check on the calculations.

When the composition of the liquid on the n^{th} plate has been established, it is possible to calculate the composition of the equilibrium vapour at any arbitrary temperature and pressure. These may be adjusted as with the feed plate, recognising that the pressure will necessarily be some 3 mm. lower than before, with a correspondingly lower temperature. Clearly this drop in temperature means a disturbance in the equimolecular flow concept, but this is slight and to some extent taken up in practice by heat losses from the column. Trial and error balances can therefore determine the vapour composition with a suitable degree of accuracy, and from this composition Σy_{an} the values for $\Sigma x_{a(n-1)}$ can be calculated. It may be noted that only approximate agreement between the calculated and stipulated pressure is required, as the relative molar fractions only change very slightly over small ranges of temperature and pressure. Repetition of this process from plate to plate up the column gives values for Σy which should converge on the arbitrary value chosen for Σy_p.

The number of plates determined by this method shows the

requirement, in perfect theoretical plates, for separation of product from feed—the "enriching" half of the column. The "perfect theoretical plate" may be defined as bringing about equilibrium between vapour and liquid, and the number of actual plates required to bring about any change of composition as compared to the number of "perfect plates" required for the same change under the same operating conditions is a measure of the "plate efficiency" of the distillation system. The plate-to-plate calculation may lead to anomalous results, in which the vapour compositions appear to diverge from the proposed product composition. This is due to the arbitrary values for reflux ratio and product composition being wrongly chosen, and suggesting a system which cannot be theoretically obtained; it is then necessary to choose other values and start the calculation afresh. This makes the method very laborious and a number of contracted methods have been suggested, of which some will be dealt with below.

The process for the enriching half of a fractionating column is a representation of conditions in a batch distillation at any one moment when the still contents and the product are specified as feed and product respectively. For the general problem of continuous working, as in Fig. III–10, it is still necessary to consider the lower "exhausting" half of the column. As the feed has been assumed to enter at the boiling-point, the number of mols of vapour rising through the column remains constant, while the liquid overflow increases from O to $O + F$. Then for this lower half a mass balance gives $V = O + F - W$. For any component A passing between the m^{th} and the $(m + 1)^{\text{th}}$ plate

$$V y_{a(m+1)} = (O + F)x_{am} - W x_{aw}$$

or
$$(O + F - W)y_{a(m+1)} = (O + F)x_{am} - W x_{aw}$$

As the value of Σx_{aw} and of W have been postulated in setting out the problem, a relation is established between liquid on the plate above and vapour from the plate below, as in the enriching half of the column. It is possible to start from the feed plate and work down the column, but it is simpler to start from the bottom plate, or "reboiler", in which vapour is generated from a liquid of the "waste" composition, and work up. In this case a series of liquid compositions converging on the feed composition will be obtained.

Now this construction is based upon initial assumptions of product and waste composition, which may or may not be correct, as

well as an arbitrary reflux ratio. If these are unsubstantiated, a great amount of calculation may be wasted, as shown below.

Example 5. *A mixture containing equal proportions of n-heptane, heptylene, toluene, and n-octane is to be distilled from a batch still, to give a product containing less than 2% of toluene–octane components. The number of perfect plates and a suitable reflux ratio are required.*

It is assumed as a basis for calculation that the first product will contain 0·60 heptylene, 0·38 heptane, 0·01 toluene, and 0·01 octane. A reflux ratio of 3 will be chosen.

Taking a trial temperature of 105° C. for the still, the vapour pressures of the pure components may be read from Fig. III–1, and the partial vapour pressures and molar fractions follow.

	P (mm.)	Px_f ($x_f = 0{\cdot}25$)	y_f ($\equiv y_{n+1}$)
Heptane . . (*a*)	890	222 mm.	0·292
Heptylene . (*b*)	1100	275 ,,	0·362
Toluene . . (*c*)	630	158 ,,	0·208
Octane . . (*d*)	420	105 ,,	0·138
		760 mm.	

As will be seen, this will give a feed plate (still) pressure of 760 mm. This still may be taken as the $(n+1)^{\text{th}}$ plate from the top of the column.

From the equation $x_n = \dfrac{R+1}{R} y_{n+1} - \dfrac{x_p}{R}$,

$$x_{an} = 1{\cdot}33\ (0{\cdot}292) - \frac{0{\cdot}38}{3} = 0{\cdot}263$$

$$x_{bn} = 1{\cdot}33\ (0{\cdot}362) - \frac{0{\cdot}60}{3} = 0{\cdot}283$$

$$x_{cn} = 1{\cdot}33\ (0{\cdot}208) - \frac{0{\cdot}01}{3} = 0{\cdot}273$$

$$x_{dn} = 1{\cdot}33\ (0{\cdot}138) - \frac{0{\cdot}01}{3} = 0{\cdot}181$$

Assuming for the next plate a temperature of 103° C.,

	P	Px_n	y_n		x_{n-1}
(*a*)	860	226 mm.	0·298		0·270
(*b*)	1050	298 ,,	0·392	from which	0·321
(*c*)	600	164 ,,	0·216		0·284
(*d*)	390	71 ,,	0·094		0·122
		759 mm.			

On continuing the calculation the following results are obtained for vapour composition:

	y_{n-1}	y_{n-2}	y_{n-3}	y_{n-5}	y_{n-7}	y_{n-8}
(*a*)	0·298	0·285	0·276	0·226	0·120	0·035
(*b*)	0·420	0·460	0·496	0·585	0·730	0·830
(*c*)	0·222	0·219	0·206	0·182	0·151	0·135
(*d*)	0·060	0·037	0·022	0·007	—	—

The hypothetical distillation values show the lower boiling heptane (a) as disappearing before the toluene (c). This is obviously wrong, and indicates either a heavier reflux of all components or a larger return of heptane. A further trial based on these altered values will be necessary, but before proceeding to this a wider consideration of the problem is required.

The most generally accepted simplification of distillation calculation is the graphical method of McCabe and Thiele, which is primarily designed for mixtures of only two pure components. These can be represented graphically on a diagram of rectangular co-ordinates, in which values on the Y axis represent molar concentrations of the more volatile compound in the vapour, and values on the X axis the corresponding concentrations in the liquid. The equilibrium curve can now be represented in the true experimental form, which may differ widely from the smooth curve suggested by Raoult's Law. Thus the curve shown in Fig. III 12 is for ethyl alcohol and water.

As in the general problem, a feed of composition x_f is to be separated into a product of composition x_p and a waste of composition x_w. The vapour leaving the top plate of the column (Fig. III 10) is all condensed, so that the composition of the liquid x_p is equal to the composition of the vapour y_p or y_1.

Consider the general equation

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{x_p}{R+1} \quad \ldots \quad (1)$$

On the diagram of Fig. III 11 this is a straight line, of slope $R/(R+1)$, passing through the point x_p, y_p and cutting the Y axis at $y_p/(R+1)$. At the top plate of the column a liquid x_1 is in equilibrium with a vapour of composition y_1 or y_p. The value of x_1 can be read off from the equilibrium curve, and substituted for x_n in the equation

$$y_2 = \frac{R}{R+1}x_1 + \frac{x_p}{R+1} \quad \text{(where } n = 1\text{)}$$

The value of y_2 thus obtained can be used to determine x_2, which is in equilibrium with y_2. Then

$$y_3 = \frac{R}{R+1}x_2 + \frac{x_p}{R+1}$$

But the points $x_1 - y_2$, $x_2 - y_3$, and similar values all satisfy the general equation (1) and the calculation above resolves itself into a stepwise construction between the equilibrium curve and the line of equation (1). This latter is defined as the " operating line " of

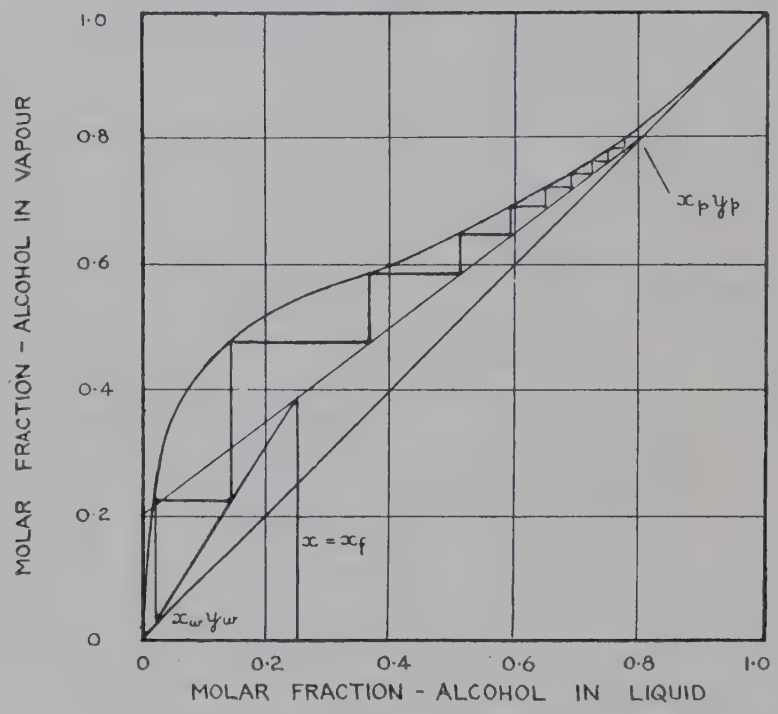


FIG. III–11.—McCABE–THIELE DIAGRAM.

the top, or " enriching ", half of the column, and represents the operation down to the plate at which the feed is introduced. With only two components in the mixture, the complete composition is fixed if the concentration of one component is known.

At the feed plate it may be assumed that the feed enters as a boiling liquid, when

$$y_{m+1} = \frac{O+F}{O+F-W}x_m - \frac{Wx_w}{O+F-W} \quad \cdot \cdot \quad (2)$$

This is another " operating line ", which passes through the point x_w, y_w and will cut the first operating line at a point where the two

line equations are simultaneously satisfied. If the co-ordinates of this point are x, y, then

$$Rx = (R + 1)y - x_p \quad . \quad . \quad . \quad . \quad . \quad (a)$$

and, as $O = RP$,

$$(RP + F)x = (RP + F - W)y + Wx_w \quad . \quad . \quad (b)$$

Multiplying (a) by P and subtracting gives

$$Fx = \{F - (W + P)\}y + (Wx_w + Px_p)$$

But on balance, $F = W + P$, and $Fx_f = Wx_w + Px_p$. Therefore $Fx = Fx_f$, $x = x_f$, and the line of intersection is independent of the value of y. It is the vertical line through $x = x_f$, and the position of intersection on this line is settled by the value of the reflux ratio chosen. It must be remembered that this construction only holds when the feed enters at boiling-point: at a later stage construction for other conditions will be given.

The second operating line can now be drawn for some specific reflux ratio. At the bottom plate a vapour is being generated in equilibrium with the "waste" liquid of composition x_w. The composition of this vapour, which may be entitled y_{m-1}, can be read from the equilibrium curve: then from the general equation of the operating line

$$y_{m+1} = \frac{O + F}{O + F - W}x_m - \frac{Wx_w}{O + F - W}$$

This determines the value of x_m, the liquid on the m^{th} plate. Again the composition of the vapour y_m follows from the equilibrium curve, and as before a stepwise construction between equilibrium curve and operating line gives the number of theoretical plates required to pass from the bottom to the feed plate. The two series of steps should meet on the feed-plate composition, but may overlap to some extent. The overlap is not of great importance in view of the approximate nature of the construction.

The variation of the operating-line positions with reflux ratio must now be considered. As more and more reflux is passed down the column, for a standard product take-off, the value of R increases and the value of $R/(R + 1)$ tends towards unity. In the limiting case, where R is infinite, the equations for both operating lines reduce to $x = y$. It will be remembered that with total reflux there is no product, no feed, and no waste, and the column operates

in a closed circuit. This system is set out in Fig. III-12a, and clearly the number of plates (steps) between a fixed waste and a fixed product is at a minimum when the reflux ratio is infinite. That is to say, the minimum height of a fractionating column required to divide the feed in some definite way is obtained when all the condensate at the top is returned to the column. On the other hand, when the amount of reflux is decreased, the slope $R/(R + 1)$ of the first operating line grows less, and the intersection with the second line draws nearer and nearer to the equilibrium curve. A

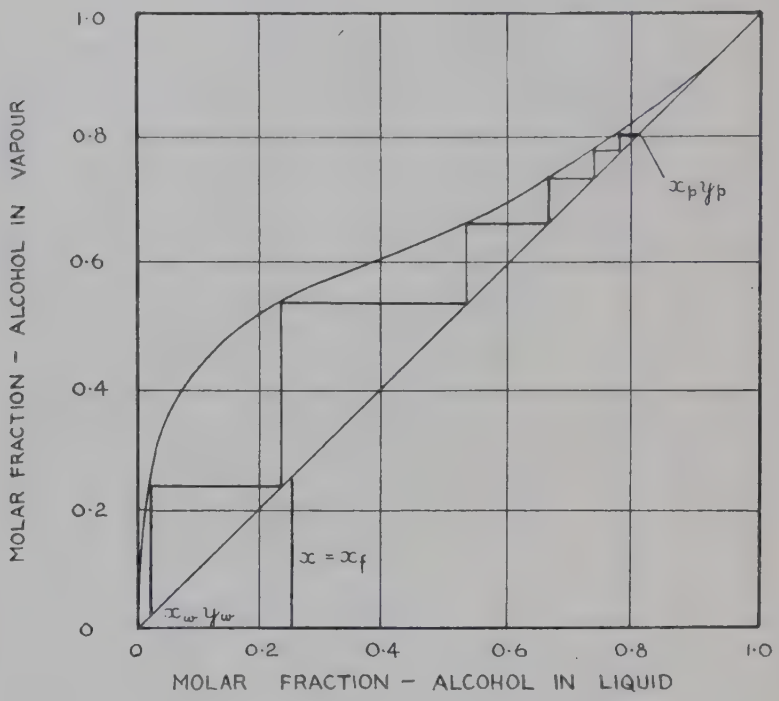


Fig. III–12a.—McCabe–Thiele Total Reflux.

limiting point is reached when the point of intersection is on the curve, or one operating line touches the curve, as shown in Fig. III–12b. The number of steps has increased as the reflux ratio has decreased, and in the limiting case the number of steps or plates is infinite, or the column required is of infinite height.

At the base of the column heat must be supplied to vaporise a portion of the descending liquid. This fraction will be determined by the value of the reflux ratio. The total amount of liquid descending is $(O + F)$, and of this $(O + F - W)$ must be re-evaporated. A factor defined as the reboil ratio S may be mentioned here. This has the value $S = (O + F)/W$, when $O + F = WS$.

Then $$O + F - W = W(S - 1)$$

Eqn. (2) may be written as

$$(O + F - W)y_{m+1} = (O + F)x_m - Wx_w$$

or $$W(S - 1)y_{m+1} = WSx_m - Wx_w$$

$$\therefore y_{m+1} = \frac{S}{S-1}x_m - \frac{x_w}{S-1} \quad . \quad . \quad (2a)$$

Now $$R = O/P \text{ and } F = W + P.$$

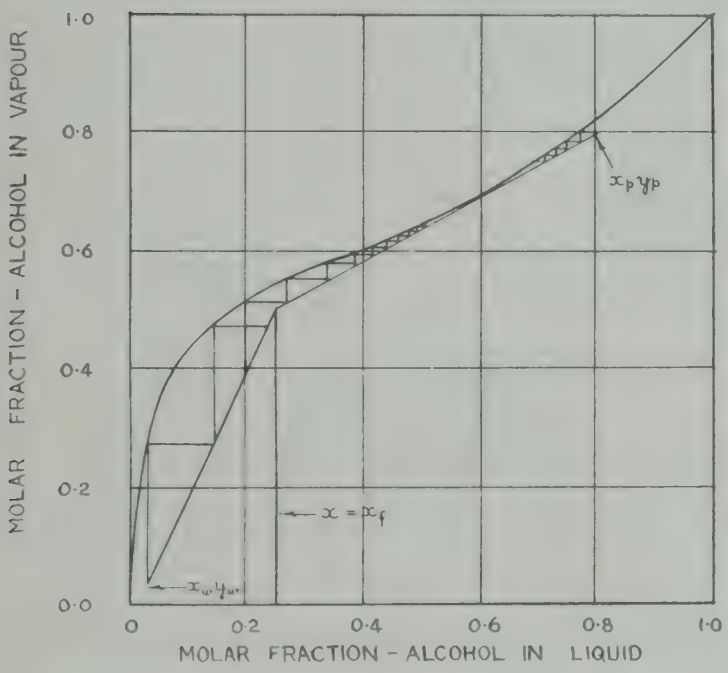


Fig. III–12b.—McCabe–Thiele Minimum Reflux.

Therefore $$S = \frac{O + P + W}{W} = \frac{RP + P + W}{W}$$

whence $$P(R + 1) = W(S - 1), \text{ or } \frac{P}{W} = \frac{S - 1}{R + 1}$$

This gives a simplified form of equation (2) and a relation between the essential quantities in a distillation.

The value of S will increase steadily as the value of O increases with a constant value for P and W, reaching an infinite value as the reflux ratio tends to infinity. Thus the shortest column requires the greatest amount of heat, while at the other extreme minimum heat requirements are given by the column of infinite height, working on minimum reflux ratio. Between the two extreme cases

shown by Figs. III-12a and 12b all practical columns have to operate, and a choice must be based on the relative costs of column construction and heat. It may be noted that the heat cost at the base of the column is matched by high costs of water supplied to the condenser; if the column is a tall one, the power required to pump water to a top condenser may be a considerable factor in choice of reflux. A balance must be drawn up for any particular case, and a typical instance is given in the table below.

Methyl Alcohol Distillation (1200 lb./hr. of 96% alcohol, feed contains 12% of alcohol, waste to contain 0·01%)

Reflux ratio	1·6	1·8	2·0	4·0	6·0
Charges in pence/hr. :					
Column charge	23·0	15·0	12·7	10·4	12·1
Building charge	3·5	2·6	2·0	2·1	2·6
Steam cost	34·0	36·3	39·6	61·7	92·0
Water cost	2·7	2·8	2·9	4·5	6·1
Total charge	63·2	56·7	57·2	78·7	112·8

Charges based on 1939 values.

The example has been greatly simplified for explanatory reasons, but it will be noted that the column charge may start to rise with very high reflux ratios, owing to the larger area of column required to accommodate the greater volumes of vapour. The actual minimum reflux ratio in the case above was 1·3, and " the minimum cost at about 1·5 times the minimum reflux ratio " is a rough rule which can be used in preliminary calculation.

This minimum reflux ratio can sometimes be calculated without the need to draw a diagram, if the mixture is known to give a regular curve for the vapour–liquid relation. Then in this case, as shown in Fig. 13a, the operating line for minimum reflux joins the two points x_p, y_p,; x_f y_f; then as the slope of the line is given by $R/R+1$,

$$\frac{R}{R+1} = \frac{y_p - y_f}{x_p - x_f}, \text{ whence } R = \frac{y_p - y_f}{y_f - x_f}$$

This calculation is very undesirable where the equilibrium curve is irregular, and where products of high purity are required; it may lead to a completely erroneous value for the minimum reflux, as shown in Fig. III–13b.

It is sometimes necessary to feed to the column a liquid which is not boiling, or which is wholly or partly in the vapour state. In this case the operating conditions in the lower half of the column are substantially modified, as the liquid downflow changes from the

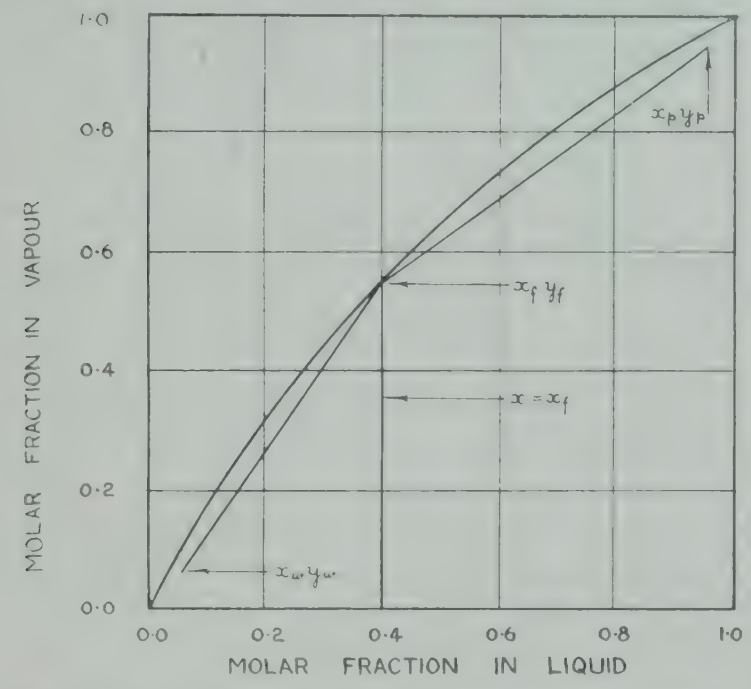


Fig. III–13a.—Effect of Calculated Minimum Reflux.

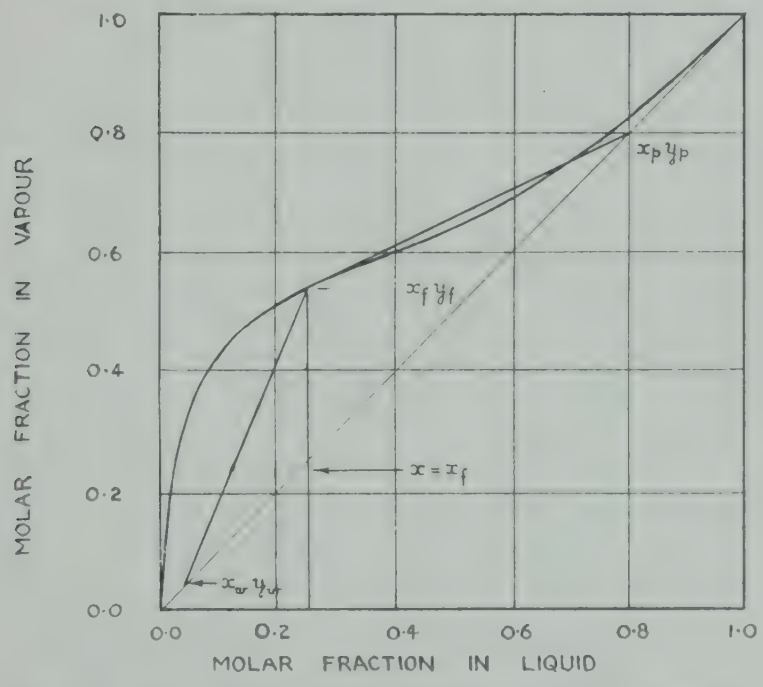


Fig. III–13b.—Effect of Calculated Minimum Reflux.

previous value of $O + F$. In general, if the ratio between the heat required to convert one mol of the feed to saturated vapour and the molar latent heat be denoted by Q, the liquid downflow will be $O + QF$. The value of Q may vary from a negative figure for superheated vapour feed, through zero for saturated vapour, to values greater than unity for a cold liquid feed.

The equation of the lower operating line will then become

$$(O + QF - W)y = (O + QF)x - Wx_w,$$

or
$$(RP + QF)x = (RP + QF - W)y + Wx_w \quad . \quad (3)$$

while the equation of the upper line remains

$$Rx = (R + 1)y - x_p \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

Multiplying (1) by P as before and subtracting from (3),

$$QFx = \{QF - (W + P)\}y + (Wx_w + Px_p)$$

or
$$QFx - Fx_f = (QF - F)y$$

Dividing this by F, and rearranging, gives for the intersection of lines represented by (1) and (3) the relation $y = \frac{Q}{Q-1}x - \frac{x_f}{Q-1}$. This equation represents a family of lines, all passing through the point $y = x_f = x$ with a slope of $\frac{Q}{Q-1}$ as shown in Fig. III–14. If the feed is a saturated vapour, $Q = 0$ and the locus of intersection is a horizontal line; if the feed is a cold liquid, Q is greater than 1 and the locus is a line sloping to the right of vertical. In the case of water a feed at 0° to a plate at 100° C. gives a value for Q of 1·185 and a line of slope (dy/dx) of 6·4.

Up to this point it has been supposed that each plate in the column will bring the rising vapour stream into equilibrium with the liquid on the plate. In actual practice this is never the case; the ratio of the change brought about by the plate to the change which a single " perfect " plate could make, is known as the " plate efficiency ". This efficiency is determined by the plate design, the mode of operation, and the characteristics of the mixture being distilled. It is not easy to give any definite value for this efficiency. In the first place, the concentration of volatile component in liquid on the plate varies irregularly, and a mean concentration is difficult to establish; even if this is obtained, the actual amount of mass

transfer, as reflected in the relative slope of operating and equilibrium lines, can affect the efficiency very considerably.* The plate efficiencies of two different designs of bubble-cap plate can therefore only be compared with the same or very similar mixtures, and are best taken for a series of plates rather than for one plate. A column with ten actual plates and a reboiler (this being equivalent to another plate) which is shown to give a separating efficiency equal to seven theoretical plates can be said to have a plate efficiency of

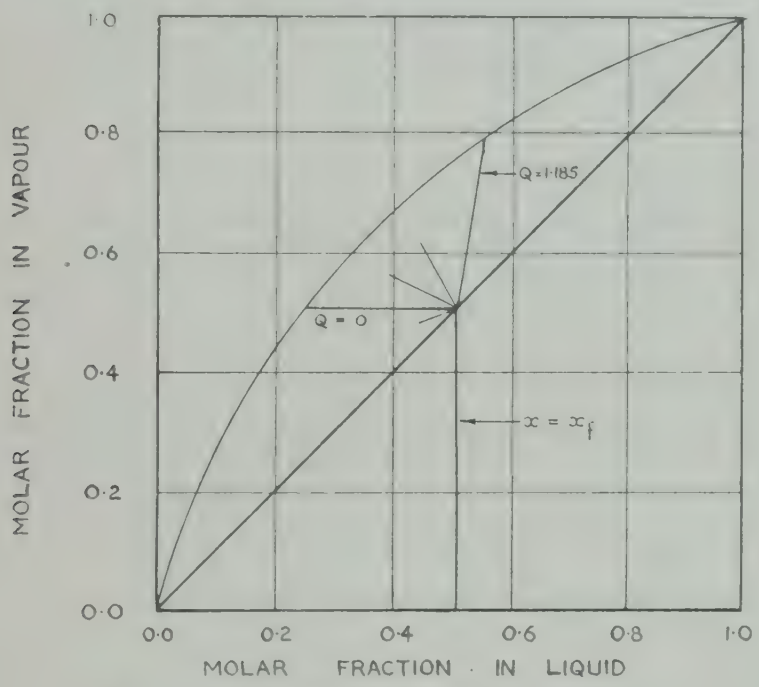


Fig. III–14.—Effect of Feed Conditions.

7/11 or 64% for that particular mixture. No very clear criterion of plate efficiency has yet been produced, but it is probable, other factors being equal, that viscous liquids in which vapour bubbles do not form readily will give low plate efficiencies. The usual plate efficiency met with in industrial practice is from 50–90%, but very low figures have been quoted† for viscous petroleum fractions, and very high figures (apparently above 100%) for large columns with wide differences in concentration.

Any binary mixture may thus be studied graphically with considerable simplicity, provided that the equilibrium curve is known. If the two components are closely related chemically, as in the case

* See, e.g., Storrow, *J.S.C.I.*, 1947, 66, 41.

† See, e.g., Gelus *et al. Chem. Eng. Prog.* 1949, 45, 602.

of two members of an homologous series of hydrocarbons, then Raoult's Law is followed closely, and the equilibrium curve can be calculated directly without need of experiment.

For a mixture of two components A and B, having molar fractions in the liquid x_a, x_b, it follows that $y_a = P_a x_a$ and $y_b = P_b x_b$, where P_a and P_b are the vapour pressures of the pure substances. But $y_b = 1 - y_a$ and $x_b = 1 - x_a$. Therefore

$$\frac{y_a}{(1 - y_a)} = \frac{P_a x_a}{P_b(1 - x_a)}$$

If the ratio P_a/P_b is defined as α, the relative volatility,

$$\frac{y_a}{(1 - y_a)} = \frac{\alpha x_a}{1 - x_a}, \text{ whence } y_a = \frac{\alpha x_a}{1 + \alpha x_a - x_a} \quad . \quad (4)$$

It has been found experimentally that the value of α does not change appreciably with composition in the case of chemically similar compounds, a generalisation which finds one form of expression in Duhring's rule as used previously in this section. The equation (4) can conveniently be used to construct an equilibrium curve for two components where the value of α is known. In the general case of a complex mixture, two of the components can be selected as " keys ", one of which is to be concentrated in the product and the other in the residue. A design for separation can now be based on graphical construction for these two components.

Example 6. *Consider the mixture studied in example* 5 *on p.* 78. *The two components chosen as keys will clearly be heptane and toluene —highest boiling component of product and lowest boiling component of residue respectively.*

The value of α *varies from* 1·42 *to* 1·46, *and a mean value of* 1·44 *will be assumed. Then the use of relation* (4) *gives the equilibrium curve of Fig. III–*15. *For the two keys, the feed contains equimolar proportions, or* $x_f = 0{\cdot}5$. *The product, as before, must have a molar fraction of toluene less than* 0·02. *The value of the minimum reflux, by drawing or calculation, is* 4·33; *this is higher than the arbitrary value of* 3, *already found to be too low. In practice a value of* 6·5 *will be chosen. Then, as will be seen from the figure,* 15 *theoretical plates will be required in the initial stages of distillation.*

It is of interest to compare this approximate graphical method with plate-to-plate calculations on the same reflux ratio basis. The following table shows the calculated liquid compositions from the feed plate

upwards, assuming the same product composition as in the previous example but a reflux ratio of 6·5:

Plate No.	*n*	*n*-2	*n*-4	*n*-6	*n*-8	*n*-10
Heptane .	0·25	0·284	0·300	0·311	0·288	0·260
Heptylene .	0·25	0·430	0·541	0·608	0·670	0·723
Toluene .	0·25	0·204	0·141	0·085	0·045	0·017
Octane . .	0·25	0·082	0·021	Neglig.	—	—

The composition of product assumed for calculation was: 0·38, 0·60, 0·01, 0·01. *The solution still needs revision, in that the calculated composition is still not converging closely on the assumed product values.*

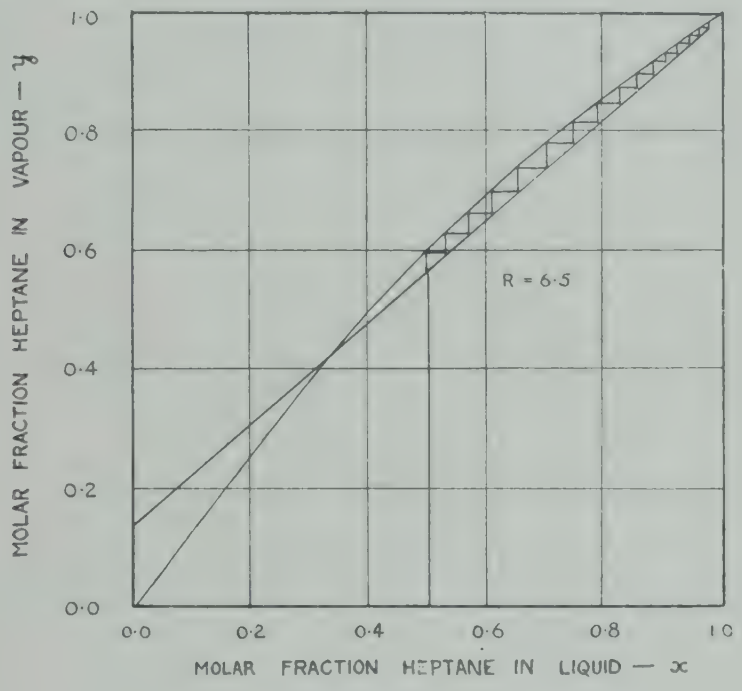


Fig. III–15.

A further trial, assuming the product composition 0·33, 0·655, 0·015, 0·00, *gave the following figures:*

Plate No.	*n*	*n*-2	*n*-4	*n*-6	*n*-8	*n*-10	*n*-11
Heptane .	0·25	0·314	0·333	0·355	0·364	0·364	0·364
Heptylene .	0·25	0·391	0·503	0·558	0·588	0·614	0·628
Toluene .	0·25	0·211	0·140	0·080	0·048	0·021	0·008
Octane . .	0·25	0·084	0·024	0·007	—	—	—

Between 10 *and* 11 *plates are required to give a liquid composition sufficiently close to the assumed value for all practical purposes.*

A study of the example above shows the value of a preliminary graphical estimate of the problem before a plate-to-plate calculation is made, but even with this indication of probable reflux ratios the plate-to-plate method is very laborious. The method has still shown only the preliminary values of reflux and plate requirements for a batch distillation, and further computations, at specified values of feed and product, will be necessary in order to complete the solution for the whole course of the distillation. For this reason the problem of multi-component distillation has been very widely studied from a mathematical angle, and a number of more or less complicated solutions proposed.* These are outside the scope of the present work, and further consideration of the mathematical side of distillation will be confined to binary systems.

For a mixture of two components, of constant relative volatility, it has been shown that the minimum reflux for a given separation is fixed by the value

$$R = \frac{y_p - y_f}{y_f - x_f}.$$

But

$$y_f = \frac{\alpha x_f}{1 + \alpha x_f - x_f}$$

and therefore

$$R = \frac{y_p - \dfrac{\alpha x_f}{1 + \alpha x_f - x_f}}{x_f\left(\dfrac{\alpha}{1 + \alpha x_f - x_f} - 1\right)}$$

When the product is nearly pure, y_p approximates to unity, and the expression for R reduces to

$$\frac{1 + \alpha x_f - x_f - \alpha x_f}{\alpha x_f - x_f(1 + \alpha x_f - x_f)} = \frac{1 - x_f}{x_f(1 - x_f)(\alpha - 1)} = \frac{1}{x_f(\alpha - 1)} \quad . \quad (5)$$

This is a value for the minimum reflux ratio in terms of relative volatility and feed composition.

Under total reflux conditions the equations for the operating lines reduce to $y_{n+1} = x_n$,

whence $1 - y_{n+1} = 1 - x_n$ and $\dfrac{y_{n+1}}{1 - y_{n+1}} = \dfrac{x_n}{1 - x_n}$

But

$$\frac{y_{n+1}}{1 - y_{n+1}} = \frac{\alpha x_{n+1}}{1 - x_{n+1}},$$

* See, e.g., Underwood, *J. Inst. Petr.*, 1946, 32, 598.

and it follows that the change in liquid composition from the n^{th} to the $n + 1^{th}$ plate is given by the relation

$$\frac{x_n}{1 - x_n} = \frac{\alpha x_{n+1}}{1 - x_{n+1}} \quad \text{or} \quad \frac{1}{\alpha} \cdot \frac{x_n}{1 - x_n} = \frac{x_{n+1}}{1 - x_{n+1}}$$

Starting from the top plate of the column, where $n = 1$,

$$\frac{1}{\alpha} \cdot \frac{x_1}{1 - x_1} = \frac{x_2}{1 - x_2}$$

But

$$\frac{1}{\alpha} \cdot \frac{x_2}{1 - x_2} = \frac{x_3}{1 - x_3}$$

Therefore

$$\frac{1}{\alpha^2} \cdot \frac{x_1}{1 - x_1} = \frac{x_3}{1 - x_3}$$

and, in general,

$$\frac{1}{\alpha^{n-1}} \cdot \frac{x_1}{1 - x_1} = \frac{x_n}{1 - x_n}$$

Taking the extra step from x_1 to x_p,

$$\frac{x_n}{1 - x^n} = \frac{1}{\alpha^n} \cdot \frac{x_p}{1 - x_p}$$

Or, in the form in which it is most easily used,

$$N \text{ (no. of plates)} = \frac{1}{\log \alpha} \cdot \log \left\{ \frac{x_p(1 - x_n)}{x_n(1 - x_p)} \right\}$$

At the bottom plate of the column $x_n = x_w$, while for exhaustive fractionation $x_p \approx 1$ and $1 - x_w \approx 1$. Then the expression reduces to

$$N = \frac{1}{\log \alpha} \cdot \log \left\{ \frac{1}{x_w(1 - x_p)} \right\}$$

This last simplification is perhaps a little unnecessary, and it is better to write

$$N = \frac{1}{\log \alpha} \cdot \log \left\{ \frac{x_p(1 - x_w)}{x_w(1 - x_p)} \right\} \quad . \quad . \quad . \quad . \quad (6)$$

The value of the relations (5) and (6) is that a rapid method is available to fix the two limits between which separation by distillation is possible. It is not usually a practical proposition to use fractionating columns of more than 50 theoretical plates, or reflux ratios higher than 10 to 1. If a test indicates plate or reflux requirements in excess of these limits, other methods of separation, or some modification of the relative volatility, must be sought.

It is possible to deal mathematically with a similar system when practical values for the reflux ratio are used. Thus Underwood* shows that

$$\left(\frac{R}{R+1}\alpha\right)^{n-1}\left\{\frac{1}{1-x_w}-\frac{(R(\alpha-1)/(R+1)}{[R\alpha/(R+1)]-1}\right\}=\frac{1}{\alpha-\alpha x_p}$$

from which the value of N may be determined as before. It must be remembered that the calculation applies to mixtures of constant relative volatility, and many of the binary systems which are of great practical importance do not give such a relationship.

In the analysis of the working of a fractionating column given above it has been assumed that the changes in liquid and vapour composition are in definite steps, at the plates. While this is correct for bubble-cap plates, it is not true for packed columns, in which a steady change in concentration occurs in both streams. As already suggested, it is possible to compare these with plate columns on the basis of the height equivalent to one bubble plate, or ideally the height equivalent to theoretical plate (H.E.T.P.). An alternative concept due to Chilton and Colburn† is that of the height of a transfer unit (H.T.U.). This transfer unit is developed from the proposition that the rate of enrichment of the vapour phase in the more volatile component is proportional to the difference between the actual vapour concentration and the true equilibrium concentration. The rate will, of course, be affected also by other factors such as extent of interface, relative phase velocities, and so on. These may be assumed as constant from top to bottom of a column for any one problem.

Then for a column section of height dH, the concentration will change by an amount $K(\bar{y}-y)$ where K is a constant, $\bar{y}$ the equilibrium concentration, and y the actual concentration in the vapour.

It follows that $$\frac{dy}{dH}=K(\bar{y}-y)$$

whence $$\int\frac{dy}{\bar{y}-y}=\int K dH$$

This expression has limits y_w, y_p and O, H.

Therefore $$\int_{y_w}^{y_p}\frac{dy}{\bar{y}-y}=KH$$

* *Trans. Inst. Chem. Eng.*, 1932, 10, 112.
† *Ind. Eng. Chem.*, 1935, 27, 255.

The integral on the left has been defined as N, the number of transfer units required to pass from y_w to y_p. The height of a transfer unit in any specific example will be given by H/N. The integral can be formulated mathematically in certain cases and solved directly, but it is simpler to choose the general form of graphical integration. For a binary system represented by a McCabe-Thiele diagram, as shown in Fig. III–16, values of $\bar{y}$ can be read from the equilibrium curve, while those of y are on the operating line; then a range of figures for $(\bar{y} - y)$, from $y = y_w$ to $y = y_p$, can be determined, and the values of the reciprocals $1/(\bar{y} - y)$ calculated. If these recipro-

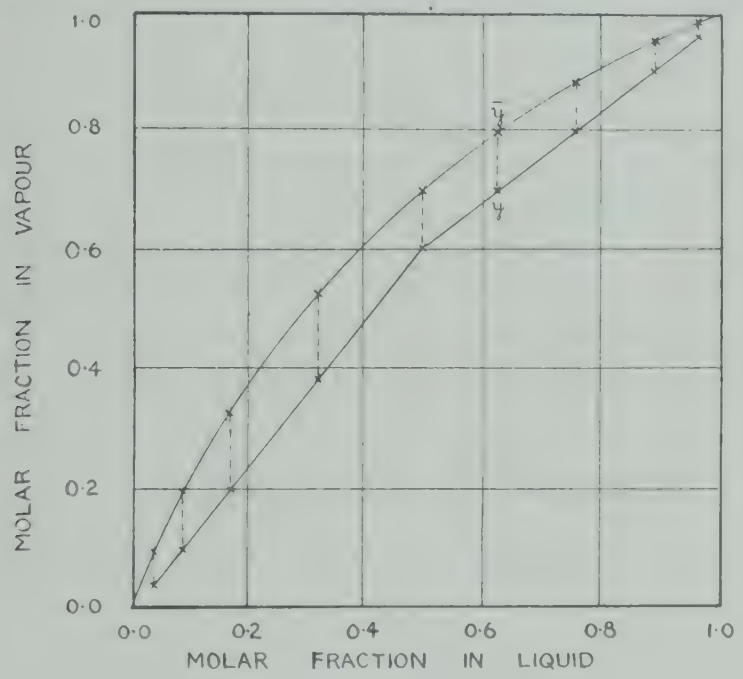


FIG. III–16.—VALUES OF $\bar{y}-y$.

cals are plotted against the corresponding values for y as in Fig. III–17, then the area under the curve will be the integral of $dy/(\bar{y} - y)$. Referring to the figure, it will be seen that the small cross-hatched area is equivalent to $5 \times 0{\cdot}1$ or $0{\cdot}5$ transfer units, and the whole area is equal to 12 transfer units.

The method is of general application in all cases of mass transfer, but does not, for distillation, appear to have any great advantage over the H.E.T.P. concept, in that widely varying results have been reported for both figures in packed column distillation. It may be assumed that a normal packed column, working with a similar mixture to a plate column, will require about 50% more height than

the plate column for the same degree of separation. This degree of separation may not be reached for wide columns unless some device for the return of liquid from the walls is used. Smoker* has described a packed column in which the outer shell of some 30 in. diameter contained seven smaller columns of 8 in. diameter through which the vapours flowed in parallel; this splitting up into several small columns may be avoided by a better choice of packing, and perhaps the glass fibre mats described by Minard *et al.*† may provide such a material.

Normal methods of distillation leave the relative volatility of the

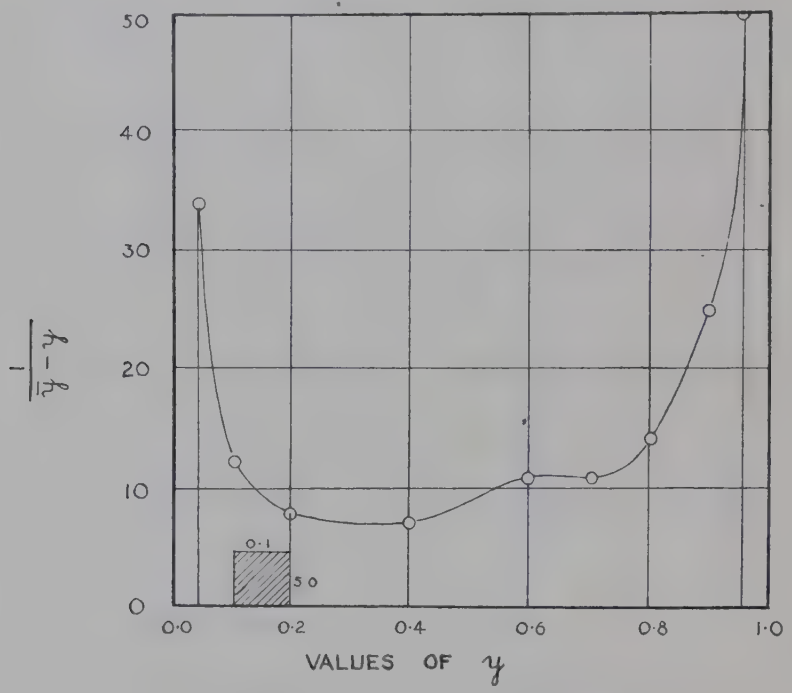


FIG. III–17.—DETERMINATION OF TRANSFER UNITS.

components of a mixture unchanged, and values of α of the order of 1·2 or less will demand high reflux ratios and very long columns. It may be possible in such cases to add another component which will alter volatility ratios to a more acceptable figure. As has already been indicated above, the partial pressure of any component above a liquid mixture can be written as $P_a = \lambda_a A x_a$, where A is the vapour pressure of the pure component and λ_a is an activity coefficient. It follows that the relative volatility (α) of the two components in a binary mixture may be defined as

* *Trans. Amer. Inst. Chem. Eng.*, 1944, 40, 105.
† *Ibid.*, 1943, 39, 813.

$\alpha = \lambda_a A / \lambda_b B$. The addition of a third component with a differential action on the activity coefficients will be to promote the volatilisation of one of the original components of the mixture at the expense of the other. This third component addition is particularly valuable when for certain compositions of the binary mixture the value of α tends to unity and a constant-boiling mixture or azeotrope is formed. The most typical and best known case of this is in the distillation of ethyl-alcohol water mixtures, where a constant-boiling mixture containing 89·4% (molar) of alcohol, with a boiling point of 78° C., represents the limit of water elimination to which ordinary distillation can go. To break down this azeotrope and to obtain absolute alcohol a number of additions have been suggested. The earliest was benzene, which increases the activity of the water and itself goes to form a ternary azeotrope, rich in water and boiling at 64·8°. On condensation the mixture separates into two phases—a water-rich fraction and a nearly anhydrous benzene alcohol phase which can be run straight back to the column for further water entrainment. The residue in a column fed with alcohol water azeotrope and a little benzene will be the absolute alcohol required. The benzene azeotrope is not ideal, as some alcohol is included, and other entrainers, such as ethyl ether under pressures above atmospheric levels, have been claimed to give the preferable binary mixture with water. This change to higher pressures illustrates another important principle, in that azeotrope formation can be profoundly influenced by alterations of temperature and pressure in the distillation system.

There is no specific need for an azeotrope to be formed in the modification of relative volatility, as can be seen from a parallel process for the production of absolute alcohol by "extractive" distillation. In extractive distillation the volatility of one component is preferentially lowered by a high-boiling addition compound, which for alcohol water mixtures may be a concentrated solution of potassium and sodium acetates. The alcohol water azeotrope is broken up, the alcohol passes over to product as the anhydrous material, and a somewhat diluted solution of the acetates flows from the bottom of the column to an evaporator for re-concentration. A similar process* for the separation of butadiene from a mixture of C_4 hydrocarbons utilises furfuraldehyde in the same way as the acetate solution. The volatility of the diene is preferentially

* *Ind. Eng. Chem.*, 1947, 39, 695.

lowered and it is discharged at the base of the column as a solution in the furfural, from which it may easily be stripped in a second column.

No definite rules have yet been suggested for the effect of added components on the relative activity coefficients. It has been suggested* that the members of a homologous series of organic compounds will all affect the vapour pressure of another compound in the same way, though with a difference in degree, and it follows from this that any constant-boiling mixture can be " broken " by the addition of another member of the series to which either component of the original mixture belongs. In the same paper methods are suggested for the choice of these additions.

The simplest constant-boiling mixture is that formed by immiscible liquids, which exert their own vapour pressures independently of each other. The vapour mixture thus formed has a constant composition directly proportional to the relative vapour pressures. The phenomenon is most frequently met with in steam distillation, which can be used to bring about volatilisation of a thermally sensitive substance at a relatively low temperature. An obvious alternative is the use of vacuum, which is in fact tending to replace the earlier process of steam distillation. Reverting to steam distillation, it may be noted that the relative values of vapour pressure alter fairly rapidly with temperature in the case of widely different substances such as water and benzaldehyde. When a steam distillation of this aldehyde is carried out at 100°, the relative vapour pressures are 60 and 760 mm.; at 130° the pressures are 190 and 2020 mm., a very considerable shift in favour of the benzaldehyde. By carrying out the steam distillation under a pressure of some 40 lb./sq. in. the steam required per pound of benzaldehyde is reduced from 2·14 to 1·8 lb.

True steam distillation implies the presence of liquid water and should be differentiated from distillation in a current of superheated steam. Here the steam acts as an inert gas in giving a pseudo-vacuum distillation, and the only advantage over any other inert gas possessed by the steam is that the vapour mixture can be more easily condensed. If superheated steam is blown through benzaldehyde at a temperature of 130°, the aldehyde will tend to give a partial pressure of 190 mm. in the vapour stream leaving, and the relative proportion of steam is settled by the absolute pressure

* *Chem. Eng. Progr.*, 1948, 44, 927.

of the mixture: if this pressure is atmospheric, then the partial pressure of steam will be only 760 − 190 or 570 mm. A distillation of this type may be the most economical of the three methods of steam vaporisation.

In concluding the section on distillation, the process of "molecular distillation" must be briefly mentioned. This process involves the removal of so much of the gas atmosphere above the liquid surface that the normal free path of molecules leaving the liquid is long enough for them to pass to a condensing surface without interference. The typical distillation unit will therefore have an evaporating and a condensing surface arranged opposite each other, and in earlier forms of molecular still they were quite close together, as in. Fig. III–18. However, as pumping efficiencies increased, and the

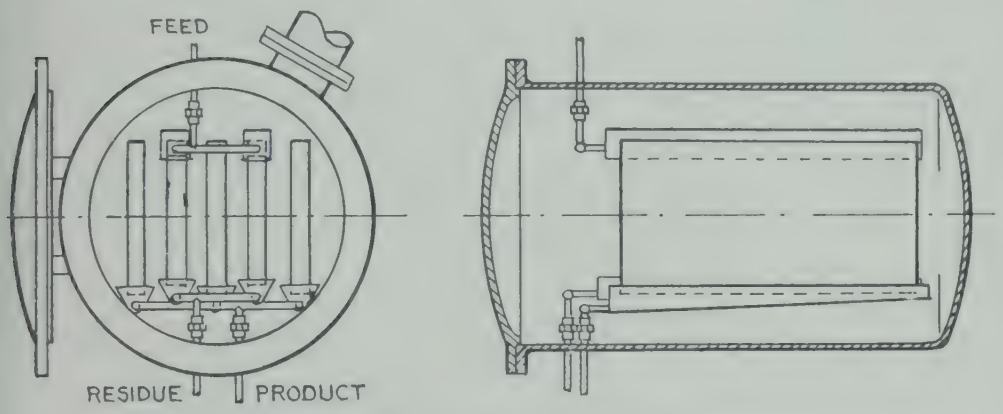


Fig. III–18.—Molecular Still.

pressure for distillation was lowered to 10^{-8} mm. or less, it was found possible to build larger units, with a considerable gap between the two surfaces. Fig. III 19 shows a typical unit of this type, in which the heated vaporising surface is a spinning disc, while the condensing surface is at a sufficient distance for the whole operation to be viewed through a glass dome. The mean free path for molecules at 10^{-8} mm. is of the order of 30 cm., so that quite a large travel of the individual molecules is possible. The rate of evaporation of a liquid of molecular weight M from a heated surface has been calculated,* on the basis of certain assumptions, to reach a maximum value of $p\sqrt{(M/2\pi RT)}$ gm. sq. cm. sec., where p is the vapour pressure in dynes/sq. cm., R the gas constant (83×10^6 dynes/cm. sec.), and T the absolute temperature. For water at 100° C.,

* *Phys. Rev.*, 1913, Vol. 2 (No. 5), 329.

this corresponds to 97 gm./sq. cm./sec., or a very much higher rate than can be obtained by direct heating. While the substances normally treated by this process have much lower maximum rates, it would appear that the principal difficulty in bulk distillation would be the supply of heat to maintain the temperature and vapour pressure constant. This is one of the reasons for the spinning disc, which gives a very thin film of liquid over a large potential heating surface.

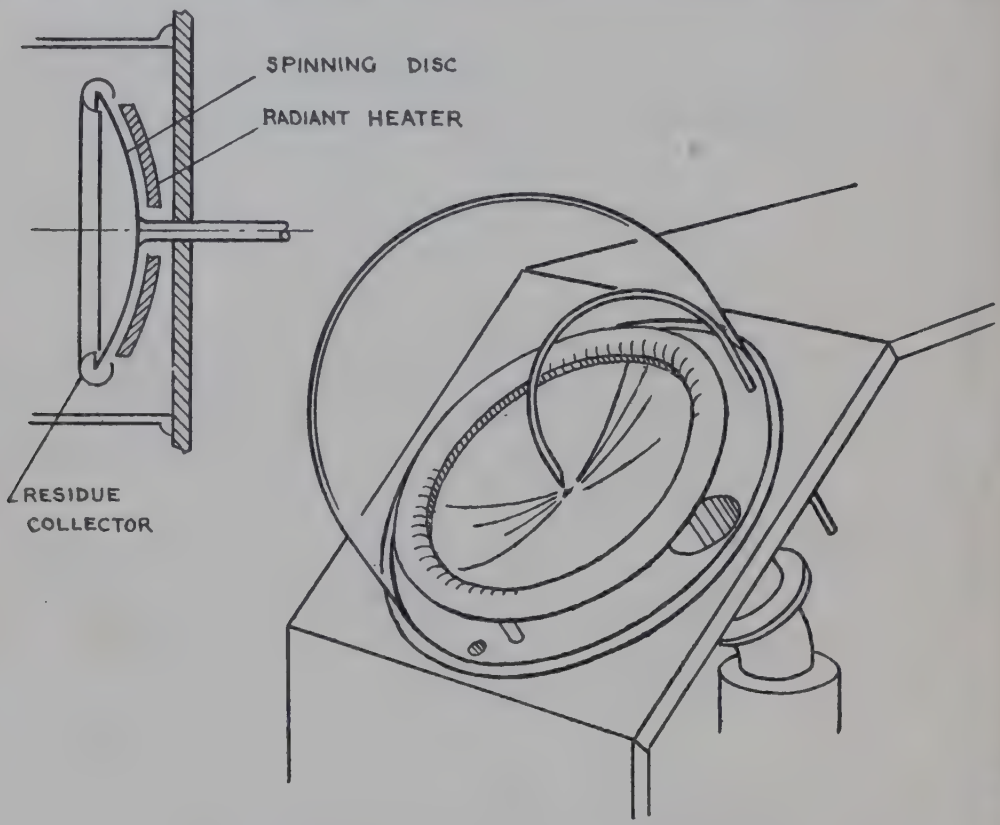


Fig. III–19.—Spinning Disc Molecular Still.

Other reasons are the need for avoidance of normal boiling with bumping and splashing, and the fact that it is the surface molecules which migrate and make it necessary to avoid covering the liquid with a skin from which the more volatile molecules have been removed. The method will deal with substances which could not be vaporised in the ordinary way, such as certain vitamin-rich oils, and is still in the development stage.*

* See, e.g., *Chem. and Ind.*, Oct. 9, 1948, Vacuum Symposium.

CHAPTER IV

GAS ABSORPTION

This is the second mass-transfer process to be considered, the third being solvent extraction of solids. As will be seen, the mathematical treatment of these two operations is practically identical, but the wide difference in actual working conditions makes it necessary to consider the two separately.

In the transfer of one component of a gas mixture to a liquid across a phase boundary, the main resistance is found to lie, as in heat

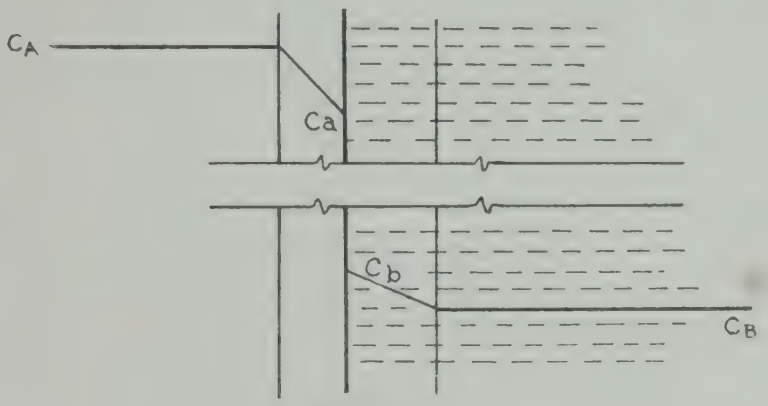


Fig. IV–1.

transfer, in the more or less stagnant films at the interface. Movement of the soluble component across these films appears to be by diffusional processes of a relatively sluggish type, and is in general proportional, for each film, to the differences in concentration existing across the film thickness. Conditions during a typical absorption may be represented as in Fig. IV-1. Here the concentration of soluble component in the body of the gas is C_A and in the liquid C_B. At the interface the corresponding concentrations are C_a and C_b. The equilibrium between a solution of a gas and the concentration of that gas in the atmosphere above the solution will vary more or less irregularly, but for most gases, over a short range of concentration, can be expressed by the simple relationship $KC_G = C_L$, where K is a constant, and C_L, C_G the concentrations in

liquid and gas respectively. At an interface between the two phases it may be assumed that equilibrium is reached instantaneously, so that, in the case set out in Fig. IV–1,

$$K \cdot C_a = C_b$$

The rate R at which the soluble component travels across the gas film may be written as

$$R = K_G(C_A - C_a) \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where K_G is a film coefficient of mass transfer, determined by the physical properties of the gas system, and giving the rate of mass transfer in suitable units (e.g. lb./hr./sq. ft./unit conctn. diff.). Similarly, as it may be assumed that the soluble component does not accumulate at the interface,

$$R = K_L(C_b - C_B) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

where K_L is the liquid film coefficient of mass transfer. Now for the liquid concentration C_B there is a gas concentration C_E in equilibrium, and then

$$KC_E = C_B$$

Substituting for C_b and C_B in (2) and rearranging,

$$\frac{R}{KK_L} = (C_a - C_E)$$

while from (1)

$$\frac{R}{K_G} = (C_A - C_a).$$

Adding these last two identities,

$$R\left(\frac{1}{KK_L} + \frac{1}{K_G}\right) = C_A - C_E$$

or

$$R = \frac{1}{1/KK_L + 1/K_G}(C_A - C_E)$$

The expression $\dfrac{1}{1/KK_L + 1/K_G}$ may be written as K_A and defined as the overall mass transfer coefficient.

Where a very soluble gas is being absorbed, K will be large, and although K_L is in general less than K_G, the product KK_L will be so large that K_A will approximate to $\dfrac{1}{1/K_G}$ or K_G. When the gas is

relatively insoluble, K will be small, and the relatively slow liquid film diffusion rate will govern the absorption. Thus, according to Hollings and Silver,* the values for the transfer of NH_3, SO_2, and H_2S from air to water are as follows:

	K	K_G	K_L	$1/K_G$	$1/KK_L$
NH_3 . .	1600	163	1·9	0·0061	0·00033
SO_2 . .	36	99	1·1	0·0101	0·0250
H_2S . .	2·5	134	0·96	0·0075	0·41

These figures are in self-consistent units, for standard experimental conditions. Just as with heat transfer, film and overall coefficients of mass transfer may vary widely with experimental conditions, as well as with the materials being absorbed. Determination of these coefficients is not difficult, but it may not be easy to simulate the conditions of large-scale industrial practice, and results will be comparative rather than absolute. A "wetted wall" apparatus described by Hollings and Silver (*loc. cit.*), is shown in Fig. IV 2. The gas passes up the tube A, down the wall of which the absorbent is flowing in an even film. It is not easy to maintain an even continuous film with some absorbents, and the author has found that a short jet of liquid may be substituted. The concentration of soluble component is determined in gas and liquid streams, and the concentration head determined from a knowledge of the equilibrium conditions. Again as in heat transfer, the effective concentration head through the transfer can be shown to be the logarithmic mean of the inlet and outlet differences in concentration (between actual gas concentration and equilibrium gas concentration). As the surface of the liquid film will be known from the dimensions of the tube (corrected for liquid film thickness) the overall film coefficient can be calculated directly.

An apparatus of this kind will show, for any particular case, the relative importance of gas and liquid films, but is not closely related to any industrial washing plant. A closer resemblance to large-scale practice can be obtained by using a small packed tower of about 6 in. in diameter, but final values for large-scale plant can only be obtained from actual industrial practice.

The design of equipment for gas absorption will clearly be greatly

* *Trans. Inst. Chem. Eng.*, 1934, 12, 49.

influenced by the relative resistance to mass transfer of the gas and liquid films. If, as is general in most industrial practice, the component to be removed is very soluble in the liquid phase, then the gas film will control, and the turbulence in the gas stream should be as high as possible. When the liquid is not a good solvent, it will be necessary to ensure that the liquid film is thin. This suggests that spray towers will be ideal in the former and bubble-cap devices

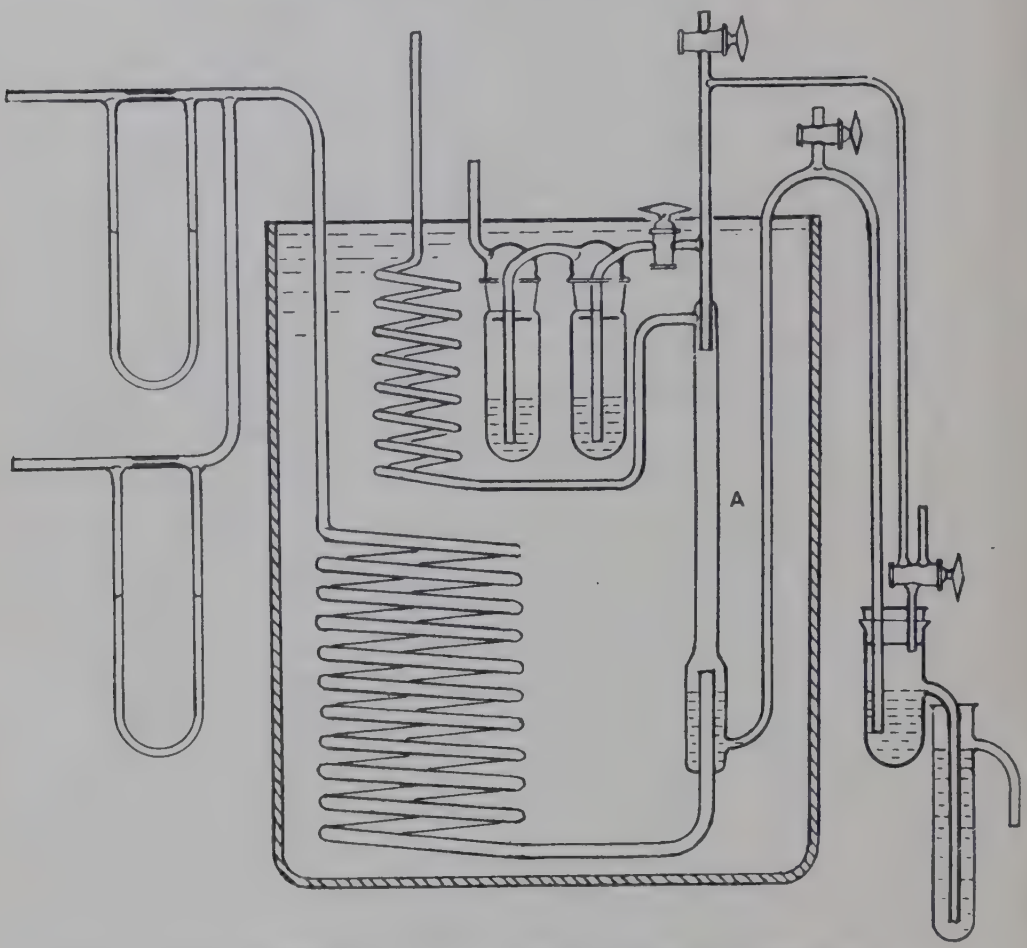


FIG. IV–2.—DETERMINATION OF MASS TRANSFER COEFFICIENTS.

in the latter system. It is seldom, however, that gas washing is carried out with a poor solvent for the component to be removed, and in any case the large amounts of liquid which would need to be circulated would make the bubble-plate design difficult. For the normal case in which the gas film controls, spray towers of various forms have been used in the coal-gas industry for many years. A typical washer of this type is shown in Fig. IV–3: a central rotating shaft passes through a series of compartments or stages, in each of

which distributor blades throw out wash liquor in a spray across the gas stream. Because of the ease with which the spray may be carried up with the gas stream, it is necessary to limit gas velocity in these washers to 2 ft. sec. at most. The liquid overflows downwards from stage to stage, while recirculating rapidly across the gas stream in each compartment. Owing to the low gas velocity, pressure drop is small, but the entrainment of liquid droplets has always been a problem. These give trouble in further operation on the outlet gas, and also interfere with the due gradation of solute concentration through the washer. A special cyclone form, suggested

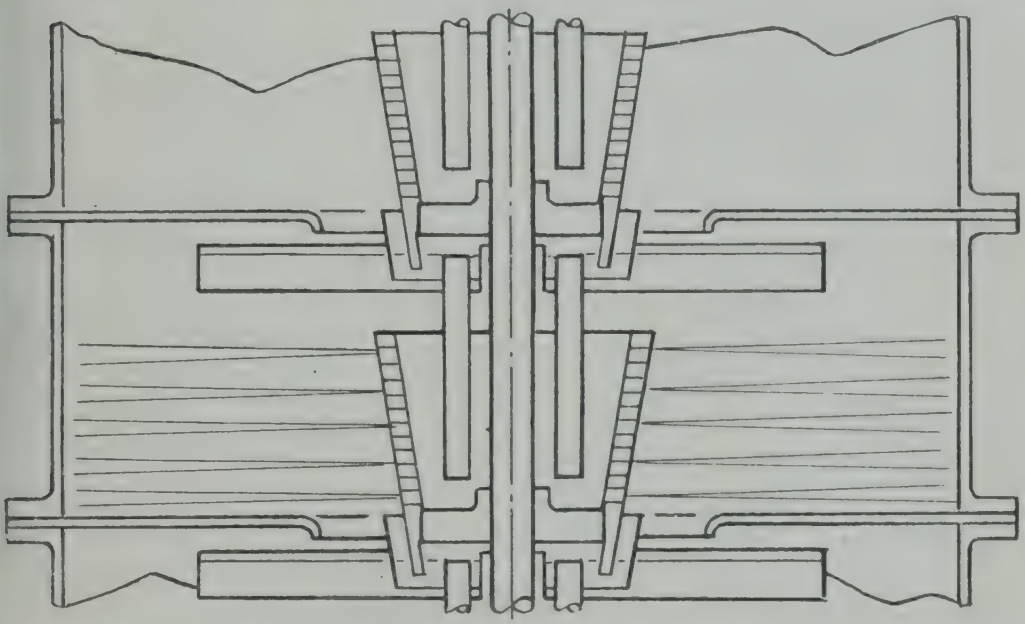


FIG. IV–3.—ROTARY SPRAY GAS WASHER.

by Johnstone and Silcox.* may do something to overcome this disadvantage. A further drawback with this type of washer is that the long central shaft is difficult to keep in good mechanical order. It would also seem that the rate of mass transfer per unit of washer volume is not so high as with the various forms of packed washing tower.†

The packed tower is the most usual form of gas-washing device; an outer shell of any suitable material is filled with a wide variety of packing, the purpose of which is to spread the solvent material over as wide an area as possible, and to split up the gas stream into a

* *Ind. Eng. Chem.*, 1947, 39, 808.

† Johnston and Bliss, *Trans. Amer. Inst. Chem. Eng.*, 1946, 42, 331.

number of small portions, all in intimate contact with a solvent wetted surface. As both liquid and gas are in motion, the packed tower should be suitable for all types of gas washing. The first and simplest type of packing was the board or slat system shown in Fig. IV-4a; the boards are arranged in bundles with spacing blocks between each board and its neighbour. A build-up of $\frac{1}{2}$-in. thick

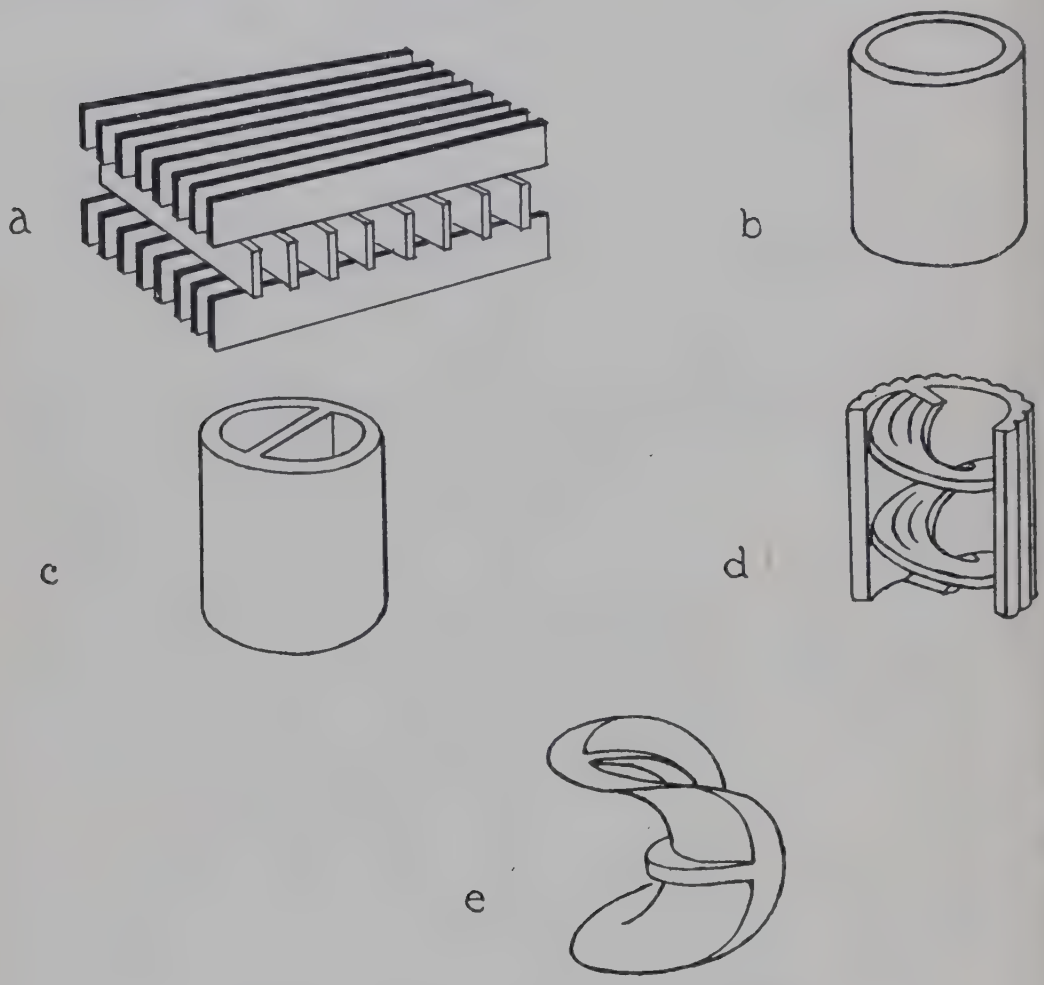


Fig. IV–4.—Packings for Absorption Towers.

boards, usually 6 in. deep, with $\frac{1}{2}$-in. spacers, will give 24 sq. ft. of potential surface per cu. ft. of packed volume. The true liquid surface will always be less than this, because of imperfect covering, and partial liquid blockage at high velocities. This is true of all types of packing, and the impossibility of determining the exact area for mass transfer in a packed tower has led to the expression of all mass-transfer figures in terms of weight transferred per unit volume of packed space. Liquid rates for good distribution in a board-packed

tower will be of the order of 100 gal./sq. ft. of tower area per hour. Gas velocities can be higher than in the spray washer—up to 5 ft. sec. at normal pressures, with a usual range of 2–3 ft./sec., again based on the total cross-section of the tower. This would mean, with board packing as described above, a linear velocity in the gas channels of just over twice this figure. The ratio of gas volume to liquid volume is about 1000 to 1 for the highest gas velocity; it has been proposed that for washing in which less liquid than this is used, other types of washers should be substituted for the packed tower.

A very large number of other packings have been devised and tested, but it is unusual to find, in large plant units, any other than the simple ring packings devised by Lessing and Raschig, although the Berl "Saddle" appears to be growing in popularity, and fibre glass packings have received some commendation. The Raschig ring, as shown in Fig. IV–4b, is a short hollow cylinder, of length equal to diameter, and is easily made up in all types of chemically resistant material. It can be filled into towers either by haphazard dumping—"random packing"—or by stacking into layers with staggered wall settings. The former is the more popular method with rings up to 1-in. diam., while careful packing is usual with rings of 3–4 in. in size. A random packing of 1-in. porcelain rings gives a maximum of 60 sq. ft./cu. ft., but results for surface will vary with the methods of packing adopted.* The hollow centre of the Raschig ring has been filled in various ways; the Lessing ring (Fig. IV–4c) is easily made from metal strip, and has a single cross partition, while the H.H. Spiral (Fig. IV–4d) forms a coil inside the outer cylinder, and is usually made in earthenware. Berl Saddles (Fig. IV–4e) have been widely used in the smaller sizes, and are claimed to give a lower pressure drop than ring packing. Fibre glass in mat form, chequer tiles with drip points, and a host of other packings have been and are being used, but it is not probable that they present any notable advance over the older forms.

The result of this wide variation in packing design has been that no general formula exists for either pressure drop or for allowable fluid velocities. It is undesirable to work a washing system close up to the limiting rate at which counter-current flow breaks down—flooding velocity condition. This limit is set by a combination of liquid and gas velocities, together with packing characteristics.

* Lobo *et al.*, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 693.

An attempt at a general correlation is given in a paper by Lobo and his co-workers,* and traces the effect of packing surfaces and voids; the relation can be represented graphically, but does not reduce to a simple mathematical form. The well-established formula of Zeisberg,† derived for air and water, can be used to give an approximate value for the pressure drop in a packed tower.

$$\text{Pressure drop (inches w.g.)} = f\frac{HV^2}{A^2}$$

where V = volume of air in cu. ft./min.,

A = cross-section of tower in sq. ft.,

H = height of tower in feet,

f is a friction factor, having values as shown in the table below.

Type of Packing	Values for $f(\times 10^7)$ with—	
	Wet (Drained) Packing	With Water at 11 lb./sq. ft./min.
6-in Quartz	60	60
2-in. „	396	433
1-in. Raschig Ring . .	345	345
3-in. Lessing Ring (dumped) .	56	63
3-in. Lessing Ring (stacked) .	54	61
Tile on Edge (approx. boards)	43	74

An alternative method may be based on the assumption that fluid flow through packed towers follows the general rules for viscous and turbulent flow, and a friction factor relation may be traced, as in Fig. IV–5. Here the dimension in the Reynolds Number is the typical size (d_0) of the packing material, and the friction factor (F) thus obtained is inserted in the formula

$$\Delta P = \frac{2F\rho v^2 A_w A_P A_L}{g d_0}$$

Here ΔP = the pressure drop per unit height, in lb./sq. ft.,

ρ = the gas density in lb./cu. ft.,

* Lobo *et al.*, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 693.

† *Trans. Amer. Inst. Chem. Eng.*, 1919, 12, (2), 231.

A_w = a wall-effect factor, approximating to unity for large towers,

A_P = a hollow-packing factor,

A_L = a liquid-effect factor.

The value of A_w is settled by the ratio of packing size to tower diameter and approximates to 1 for values of the ratio less than 0·05. The value of A_P varies more widely, but determinations by White* show figures of 0·2 to 0·3 for $\frac{1}{2}$-in. to 2-in. Raschig rings. The value

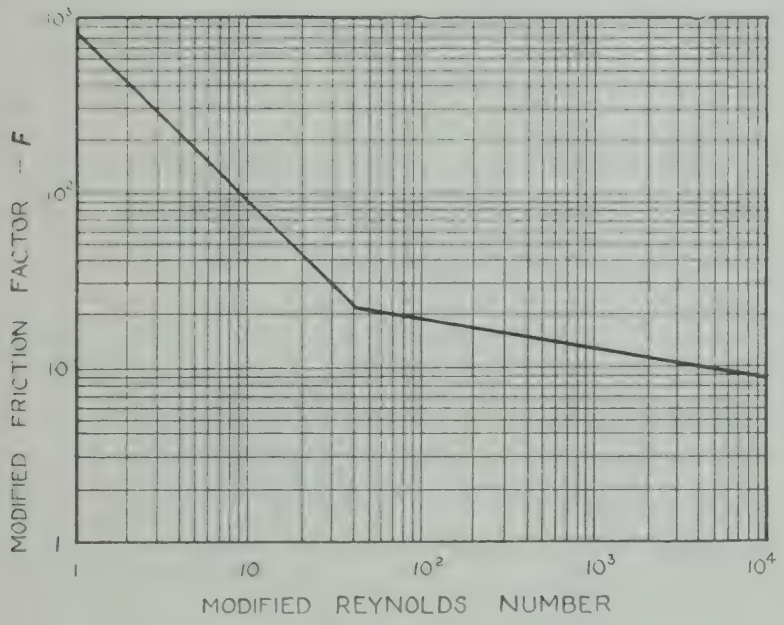


Fig. IV–5.

(White, *Trans. Amer. Inst. Chem. Eng.*, 1935, 31 (2) 390.)

of A_L varies from 1·5 to 2 with 1-in. ring packings and liquid velocities from 100 to 6000 lb./hr./sq. ft.

Example 1. *A tower 3 ft. in diam. and 30 ft. high, packed with 1-in. Raschig rings, is scrubbing air at a rate of 25,000 cu. ft./hr., using water at a rate of 5000 lb./hr. Calculate the pressure drop over the tower.*

The gas velocity (v) *is 1 ft./sec., and the kinematic viscosity* (λ) *of air at 15° C. is* $1{\cdot}6 \times 10^{-4}$ *F.P.S. units.*

Then
$$\frac{vd_0}{\lambda} = \frac{1 \times 0{\cdot}0875}{1{\cdot}6 \times 10^{-4}} = 553$$

* *Trans. Amer. Inst. Chem. Eng.*, 1935, 31, (2), 390.

From the chart of Fig. IV–5, $F = 15$.

$$\textit{Then} \quad \Delta P = \left(\frac{2 \times 15 \times 0{\cdot}075 \times 1^2 \times 0{\cdot}2 \times 1{\cdot}6 \times 30}{32{\cdot}2 \times 0{\cdot}0875}\right)$$

$$= 7{\cdot}7 \textit{ lb./sq. ft., or } 1{\cdot}5 \textit{ in. w.g.}$$

It is interesting to compare this with a calculation based on Zeisberg's formula.

Water is flowing at 11·8 *lb./sq. ft./min., so that f will be* 345×10^{-7}. *The gas velocity is* 417 *cu. ft./min., in a tower of* 7·05 *sq. ft. section.*

The pressure drop is therefore

$$\frac{(3{\cdot}45 \times 10^{-5}) \times 30 \times 417^2}{7{\cdot}05^2} = 3{\cdot}6 \textit{ in. w.g.}$$

The variation between these two solutions for the same problem is an indication of the inadequacy of any general formula, and suggests that a general safety factor should be allowed in the construction of pumping devices for working these towers.

The packed tower requires for efficient working that the amount of liquid circulating shall not be too small. It is probable that liquid distribution becomes very poor for rates of less than 500 lb./sq. ft./hr., assuming that the liquid has the general physical characteristics of water or dilute solutions. This has been crystallised in the rule that for liquid : gas ratios (Q) less than 0·001, a packed tower should not be used, and some other type of washer substituted. The bubble-cap system, so widely used in distillation, can be adopted, and is well suited to absorptions in which the liquid film is important. These are uncommon, and in addition the high pressure drop means a very large pumping cost when large volumes of gas are involved. A twelve-plate column, giving the equivalent of about 8 " perfect theoretical stages ", will involve a pressure drop of at least 12 in. w.g., whatever the gas velocity. As will be seen later, this is much higher than for an equivalent contact in other types of washer. Nevertheless, the bubble-cap column has been used for gas absorption, especially in the petroleum industry; an attempt to assess the performance of a number of large columns in this work was made by E. G. Ragatz and J. A. Richardson,* and it appears that " plate efficiency is diminished very considerably by the large amounts of almost inert gas present ".

* *Oil and Gas Journ.*, 1946, 45, (29), 89.

The general operating details of these bubble-cap plates have already been covered in the chapter on distillation.

A more suitable design is the film washer, most frequently made up in the form shown in Fig. IV-6. A horizontal cylinder is divided into compartments by suitable baffles, so that the gas flows through each stage in turn. A central axle rotates a series of brushes, rather like chimney brushes in plan, but much larger—up to 10 ft. in diameter. These brushes dip into the wash liquid, which is flowing from stage to stage counter-current to the gas, and carry up into the gas space sufficient liquid to keep the brush surfaces wet until they re-dip into the liquid. The brush can carry small troughs, (not shown

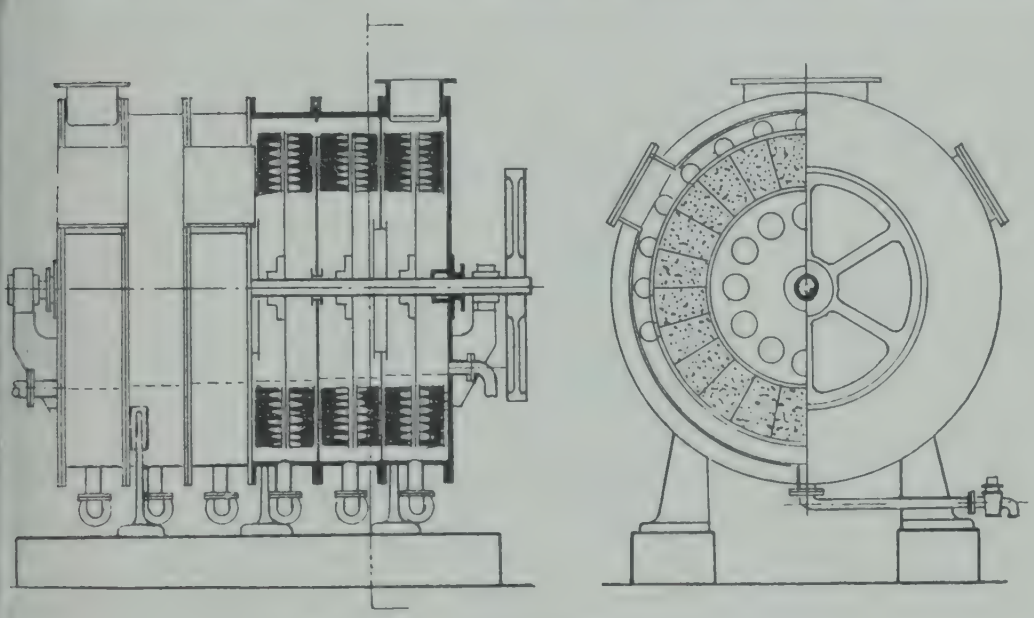


Fig. IV-6.—Rotary Film Washer.

in the figure), which improve the wetting and allow the liquid level in the washer to be well below the middle of the cylinder. As a result, practically the whole of the washer volume is active space for gas absorption. The area exposed by the brushes will vary with the construction, but for large washers is at least 25–30 sq. ft./cu. ft. The brushes can be made of stiff wire, thin metal or wooden sheets, or thin wooden rods, and as all the liquid is on a solid surface, possible gas velocities are high. Linear velocities over the brush surfaces of 8 ft./sec. have been quoted for ammonia washers operating on coal gas. The brushes are rotated at quite slow speeds—2 to 4 r.p.m.—and the power requirements for driving are very low. The pressure drop in passing such a washer is about 0·1 in. w.g. per

compartment. A washer 8 ft. in diameter and 16 ft. long, with eight compartments, treated 167,000 cu. ft. of gas per hour, with a pressure drop of 1 in. w.g., in removing ammonia from coal gas.

When the amount of liquid to be circulated is very small, or when it does not readily adhere to the brush fibres, a " static " film washer can be used in which the liquid is pumped continuously over the surfaces. There is no need to make up this unit in tower form, or even in continuous sections, and it is usually built in the form of a rectangular horizontal block, as shown in Fig. IV–7. As the liquid is circulating rapidly in each stage, there is no need to adopt counter-

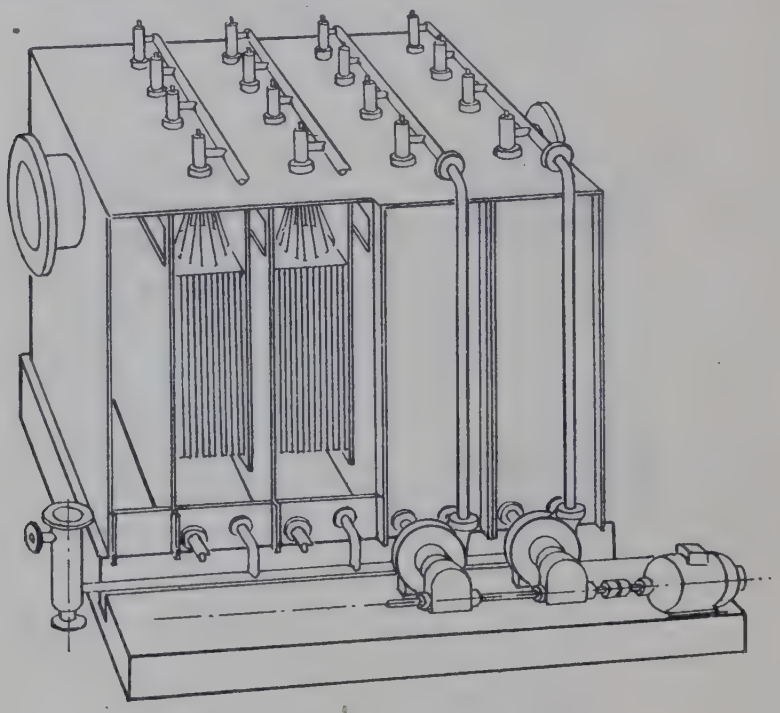


Fig. IV–7.—Static Film Washer.

current flow, and both liquid and gas can flow down over the brush fibres in each compartment. A small side-current from each pump to the next compartment produces the required liquid flow, and this positive flow is counter-current to the gas stream. These " static " washers are tending to replace the rotary type in all fields.

Now all these washing units have gas and liquid streams flowing in opposite directions. It may be assumed that such a system can be divided into a number of stages, in each of which the two streams reach either equilibrium or some definite point on the way thereto. This concept may be compared to the idea of a H.E.T.P. in distillation, and is nearly approached, in real operation, by the individual

stage in a film washer. Here the liquid remains approximately constant in composition in each stage, and changes sharply in composition from stage to stage. The gas composition, on the other hand, changes steadily throughout.

Consider (Fig. IV 8) a washing system of n stages, in which the concentration of a soluble component is reduced from y_n to y_0, by washing with a fixed amount of non-volatile solvent. If it is assumed that the gas volume does not change appreciably in washing, an assumption which is nearly true in most cases, then the ratio of liquid volume to gas volume will be constant throughout at

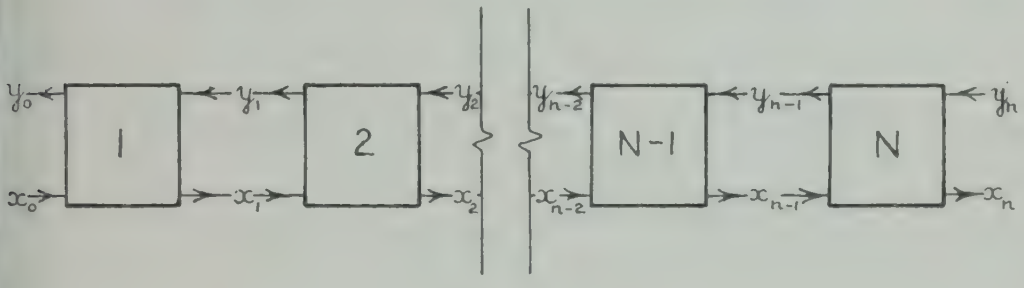


FIG. IV–8.

some value Q. Then the final concentration of the liquid leaving the n^{th} stage and denoted x_n will be given by

$$x_n = \frac{y_n}{Q} - \frac{y_0}{Q} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

Now if each stage is large enough for the gas to come into perfect equilibrium with the liquid in that stage, then the liquid x_n will be in equilibrium with the gas leaving that stage, of composition y_{n-1}. If it is assumed that Henry's Law holds, then the relation between these last two is given by

$$x_n = K y_{n-1} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

The relations expressed by equations (3) and (4) will clearly hold for all stages of the system, with appropriate concentration figures, and may be represented graphically as in Fig. IV 9. In any real system the line of equation (3) will lie to the left of the line of (4), and the distance between them is a measure of the concentration head causing mass transfer. If the lines cross, this concentration head is reversed, and the soluble component will pass from the liquid to the gas—stripping instead of absorbing.

In the n^{th} stage of the washer, the concentration in the gas falls

from y_n to y_{n-1}, in contact with a liquid of concentration x_n, a change represented by the first vertical step in Fig. IV–9. The value of x_{n-1} is given by $x_{n-1} = \frac{y_{n-1}}{Q} - \frac{y_0}{Q}$, and can be read off at the end of the first horizontal step of the figure. Change in gas concentration in the second stage is given by the second vertical line, and the continuation of this downward vertical construction gives the number of theoretical stages required before the terminal concentration y_0 is reached. The slope of the line $x = \frac{y}{Q} - \frac{y_0}{Q}$ is

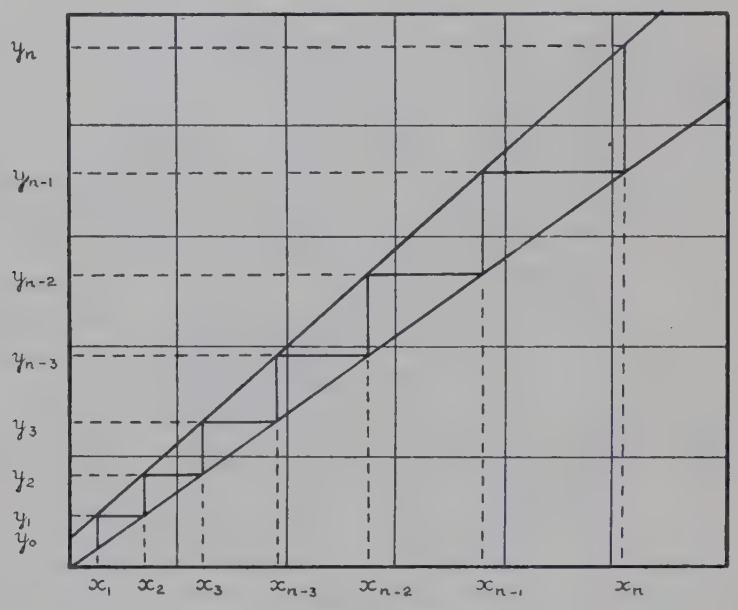


FIG. IV–9.

settled by the value chosen for Q. This arbitrary ratio of liquid–gas volume has no upper limits, but the lower limit, as with minimum reflux ratio in a distillation problem, is rigidly fixed. As will be seen from Fig. IV–10, it is that value of Q which causes intersection of the two " operating lines " at the point y_n, Ky_n.

Then $$Ky_n = \frac{y_n}{Q} - \frac{y_0}{Q}, \text{ or } KQ = 1 - \frac{y_0}{y_n}.$$

The ratio y_0/y_n is usually small, and may be defined as the slip (S) of the washing system. It follows that $KQ = 1 - S$, or $Q = (1 - S)/K$. This represents the limiting value of Q in terms of the slip and the equilibrium constant. Smaller values of Q cannot give removal of the soluble component between the limits of y_n, y_0

no matter how many stages are used. It will be noted from Fig. IV–9 that in the hypothetical case represented, between seven and eight stages are required. It will clearly be possible to adjust either the slip or the value of Q until an exact number of stages are shown, but there seems little point in this, as in practice eight stages would be allowed, and the method is in any case an approximate one.

Now in each of the stages the gas is supposed to come into equilibrium with the liquid. The rate of mass transfer in these washing operations has been shown earlier to be proportional to surface, mass transfer coefficient, and concentration head $(C'_G - C_E)$. In the first stage (Fig. IV-9) the initial value of $(C'_G - C'_E)$ is clearly

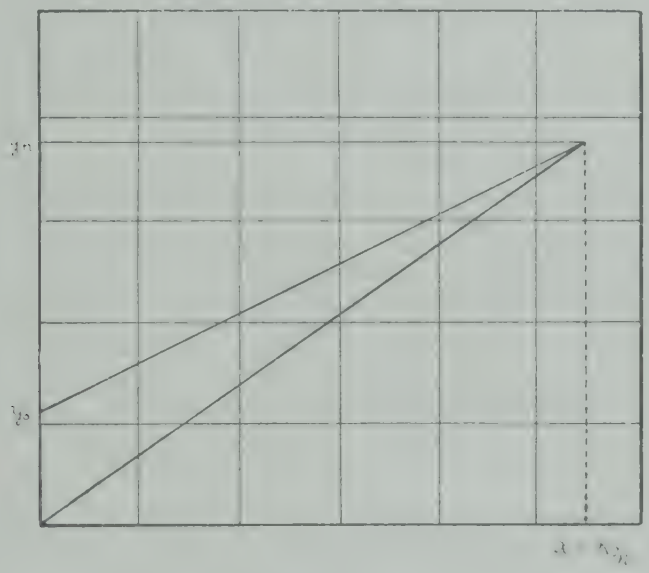


Fig. IV–10.

$(y_n - y_{n-1})$, and this drops to zero as the gas comes into equilibrium. The effective mean concentration will be the logarithmic mean of these values, as in the parallel case of heat transfer, and this will be infinitely small. That is to say, the surface required for perfect equilibrium will be infinite, and the perfect stage has no real existence. This difficulty can be overcome by assuming that in each stage the change in concentration in the gas is some 70% of the complete change to equilibrium. This 70% is an arbitrary value, but is in fair agreement with practical working figures. Then working conditions in the washer can be represented by Fig. IV–11, where each vertical step is interrupted at a point $\frac{7}{10}$ths of the way down. Then the change in concentration in the first stage is from

y_n to y_{n-1}, with a concentration head of $y_n - y_E$, altering to $y_{n-1} - y_E$ as the gas leaves the stage. These values are both finite, and a definite value for the logarithmic mean concentration head can be obtained. Now if the value of the overall mass transfer coefficient has been obtained experimentally for the conditions existing in the washer, then the surface required for mass transfer at each stage can be calculated.

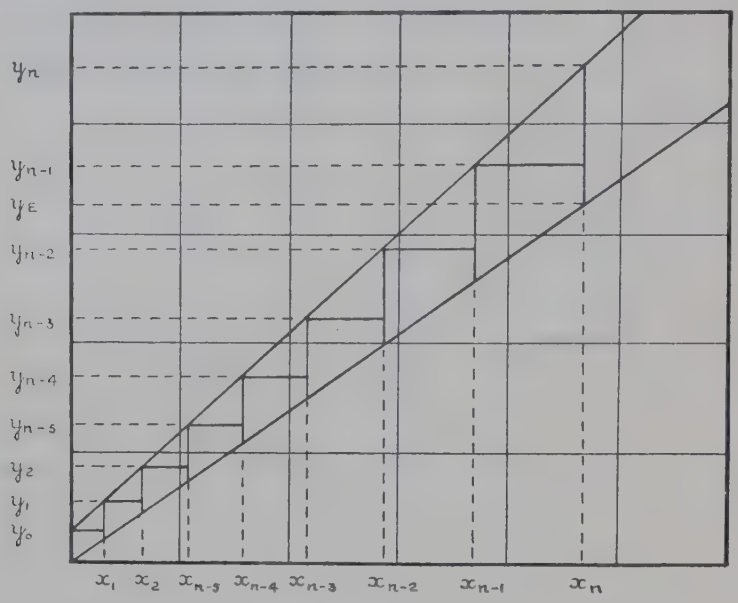


FIG. IV–11.

Example 2. *A rotary gas washer is required to treat* 1,000,000 *cu. ft. of coal gas per hour and to reduce the ammonia concentration from* $1{\cdot}0 \times 10^{-3}$ *lb./cu. ft. down to* 1×10^{-4} *lb./cu. ft. A value for* K_G *has been experimentally determined at* 500 *lb./sq. ft./hr./lb. cu. ft. conctn. head. The value of* K *is approx. constant at* 3000, *and it is proposed to use three times the theoretical minimum amount of water.*

The slip S *of the washer is* $\frac{1}{10}$ *or* 0·1, *and from this the minimum value of* $Q = \dfrac{1 - 0{\cdot}1}{3000}$, *or* 3×10^{-4}. *The value of* Q *chosen will be* 9×10^{-4}. *The final concentration* x_n *in the liquid will be given by*

$$x_n = \frac{(1 \times 10^{-3}) - (0{\cdot}1 \times 10^{-3})}{9 \times 10^{-4}} = 1{\cdot}0 \text{ lb./cu. ft.}$$

The operation of the washer can now be represented as in Fig. IV–12. Between three and four " 70% *" stages will be required ; as before an*

adjustment of slope or slip would give an exact number of stages, but this is not considered necessary.

In the first stage of the washer, the conctn. head, initially $6{\cdot}7 \times 10^{-4}$, *is reduced to* 2×10^{-4} *lb./cu. ft. This gives a logarithmic mean concentration head of* $3{\cdot}9 \times 10^{-4}$ *lb./cu. ft. The amount of ammonia to be removed is* $4{\cdot}7 \times 10^{-4} \times 10^{6}$ *lb. or* 470 *lb./hr.*

By definition $470 = 500 \times 3{\cdot}9 \times 10^{-4} \times S$, *where S is the surface*

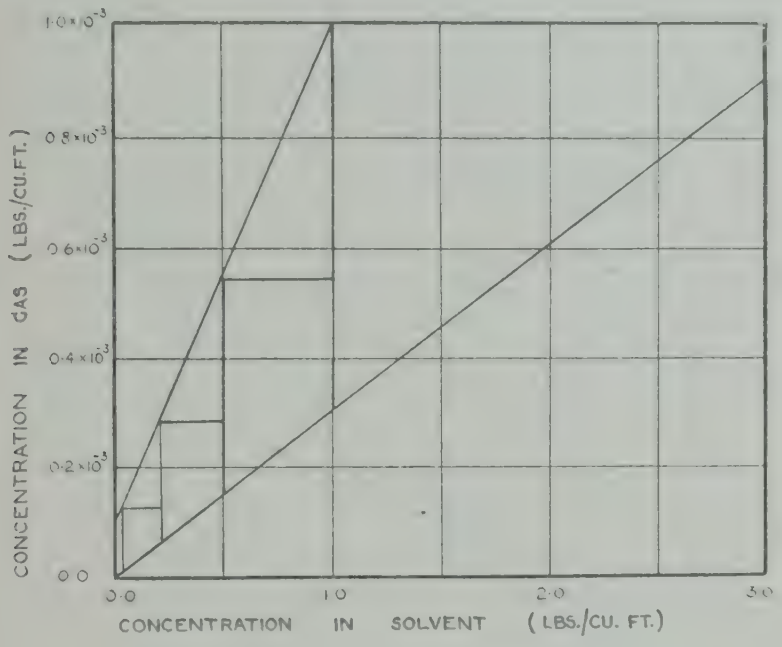


Fig. IV–12.

required in the stage. Then $S = 2400$ *sq. ft. Similarly for stages* 2 *and* 3

Stage	*Amt. Removed*	*Conctn. Head*	*Surface*
2	260 lb.	$2{\cdot}2 \times 10^{-4}$	2400
3	142 lb.	$1{\cdot}18 \times 10^{-4}$	2400

This equal value at each stage could have been predicted from similarity considerations. A fourth stage would be added to give a reasonable working margin.

Where the "operating lines" are straight, as in the figure, it would be possible to calculate the amount of surface required for the total transfer by consideration of the mean driving force throughout the washer. This surface would agree with a stage-by-stage calculation if an exact number of stages was selected by trial and error; there would be no indication of the number of stages required, a

serious flaw from a design point of view. If the equilibrium curve is not straight but curved, as in Fig. IV–13, indicating that Henry's Law does not hold, then the calculation must be carried out in stages. This is particularly necessary where the gas film is not the only factor governing mass transfer. Considering the diagram of Fig. IV–13, when a very soluble gas is being washed out of a mixture, the gas film controls, and interfacial conditions approximate to the point A, with A–C representing the driving force. Where a relatively insoluble gas is being removed, the liquid film controls, interfacial conditions approach the point B', and the driving force is proportional to $B' - C'$. If both resistances are to be considered,

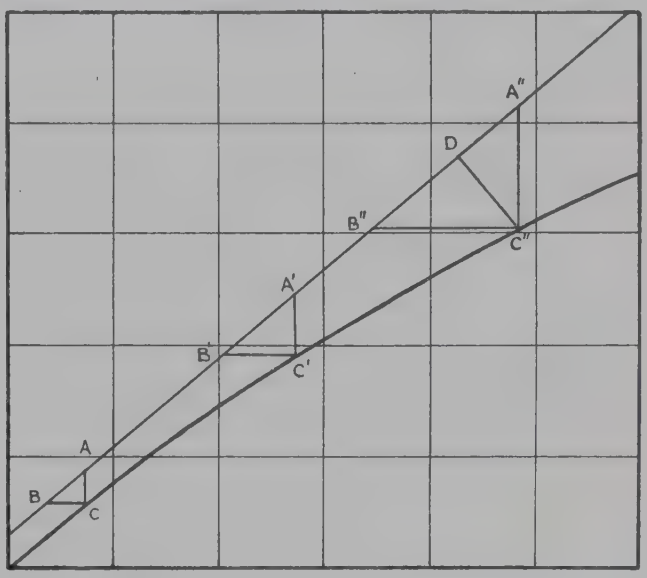


Fig. IV–13.

interfacial conditions may approximate to some point D, and the driving force to some length D–C'', the slope of which is determined by the relative resistances of the two films. When the " operating lines " are straight, all these lines representing driving force have some regular relation to each other, and any one can be used, with suitable experimental values for the mass transfer coefficient. If there is any irregularity in the lines, this is no longer true, and the correct driving force representation must be chosen. However, all practical cases of gas washing use good solvents for the component to be removed, and the construction will be based on gas concentrations and on the vertical lines of the diagrams above.

The division of the system into stages makes for an unreal picture

of washing in a packed tower, just as in the parallel case of distillation. It has therefore been suggested that the transfer unit concept should be applied to gas washing.

Consider a tower of unit cross-section, through which G mols of gas pass in unit time. Then if the amount of surface per unit volume is S, and the tower is divided into slices of height dH,

$$-Gdy = K_G S dH(y - y_E)$$

It follows that

$$-\frac{dy}{y - y_E} = \frac{K_G S}{G} dH$$

Integrating this expression between the limits O, H and y_n, y_0, where H is the tower height, and the gas enters with a solute concentration

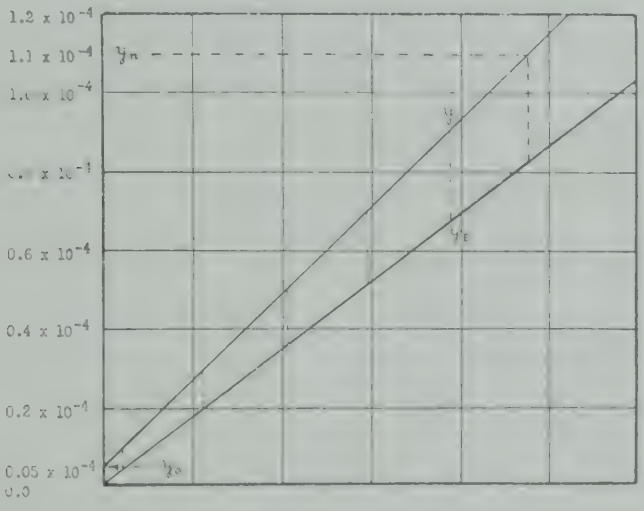


Fig. IV–14a.

y_n at the tower base and leaves with a concentration y_0 at the top, gives

$$\int_{y_0}^{y_n} \frac{dy}{y - y_E} = \frac{K_G S}{G} H$$

As in distillation, the left-hand integral is defined as N, the number of transfer units to which the tower is equivalent. Then the H.T.U., or height of transfer unit, will be given by H/N. The units for gas concentration can be given any self-consistent form, as both sides of the integrated equation are dimensionless. The integral can now be determined graphically, as in Fig. IV–14a and 14b.

For typical operating conditions, as shown in Fig. IV-14a, the gas concentration is reduced from $1{\cdot}1 \times 10^{-4}$ down to $0{\cdot}05 \times 10^{-4}$. From this figure the following table may be drawn up :

y	y_E	$y - y_E$	$1/(y-y_E)$
$1{\cdot}10 \times 10^{-4}$	$0{\cdot}72 \times 10^{-4}$	$0{\cdot}38 \times 10^{-4}$	$2{\cdot}64 \times 10^{4}$
0·90 ,,	0·58 ,,	0·32 ,,	3·14 ,,
0·70 ,,	0·45 ,,	0·25 ,,	4·00 ,,
0·50 ,,	0·31 ,,	0·19 ,,	5·27 ,,
0·30 ,,	0·18 ,,	0·12 ,,	8·34 ,,
0·10 ,,	0·035 ,,	0·065 ,,	15·40 ,,
0·05 ,,	0·00 ,,	0·05 ,,	20·00 ,,

The value of $1/(y - y_E)$ can now be plotted against y as in Fig. IV–14b. Then the area under the curve represents the

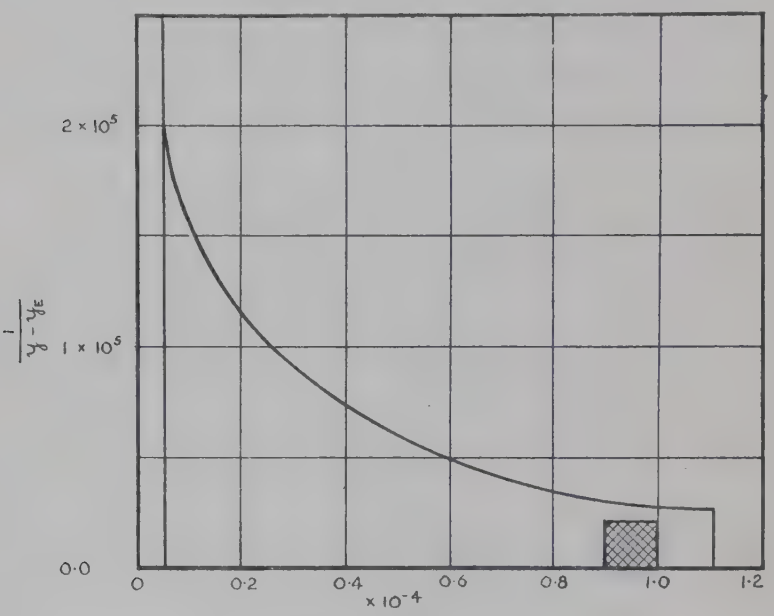


Fig. IV–14b.

integral and may be computed by counting squares. The cross-hatched square has an "area" of $(0{\cdot}1 \times 10^{-4}) \times (2 \times 10^{4})$ or 0·2 of a transfer unit, and the total area contains the equivalent of 32·75 squares, or 6·55 transfer units are represented. This treatment is identical with that given for distillation problems, but a warning must be given that mass-transfer rates, as between a gas stream and a solvent for one component of the gas, are much lower than those between saturated vapour and boiling liquid. Then the H.T.U. for

mass transfer will be much greater than that which can be obtained with the same type of packed column in distillation practice.

As with the graphical step method, the H.T.U. computation can be based on liquid concentration heads, or on some intermediate form, but is best referred to gas concentrations. The solution of any problem will depend upon the experimental determination of the H.T.U. for any particular type of column, just as the stepwise method depends upon determination of the mass transfer coefficient. This latter method has the advantage of showing where the absorption is actually occurring in any type of washer, and by presenting a picture of the theoretical change in gas composition makes it easy to check the practical working of a plant.*

The extent of the liquid surface actually in contact with a gas stream is always doubtful, especially in such units as packed towers with dumped packing. It may therefore be simpler to compare absorption efficiencies on the basis of a volume mass transfer coefficient — weight transferred/hr./cu. ft./unit concentration head. This coefficient, denoted by K_{G_a}, has been given in a very wide range of units, and an arbitrary and universal choice would be desirable. In the rest of this work the value of K_{G_a} will be given in pound mols/hr./cu. ft. per unit conctn. head in atm. in gas phase. As it is supposed that gas-film conditions control, it can be anticipated that values of K_{G_a} will vary as some power of the gas velocity. Thus Scheibel and Othmer† correlate a large number of experimental values on the basis of a formula including $G^{0.8}$, where G is the gas velocity in lb./hr./sq. ft. of washer cross-section. A general equation for the absorption of ammonia from an air stream by water has been proposed by Sherwood,‡ and can be expressed as $K_{G_a} = \phi G^{0.8}$, where ϕ varies from 0·057 to 0·147 according to the type of packing used. The results are in general agreement also with figures for SO_2 absorption quoted by Sherwood (*loc. cit.*) when allowance is made for the relative rates of diffusion of NH_3 and SO_2 molecules in air.

The actual diffusitivity (D) for typical gases in air varies from 0·06 for heavy molecules such as ethylene tetrachloride to 0·19 for H_2O, and values for K_{G_a} have been shown experimentally to be proportional to $D^{0.56}$. When the actual gas medium changes, this

* See, e.g., Bagg, *Trans. Inst. Chem. Eng.*, 1934, 12, 223.
† *Trans. Amer. Inst. Chem. Eng.*, 1944, 40, 611.
‡ " Absorption and Extraction ", p. 178.

diffusivity will vary inversely as the square root of the gas density.* It is doubtful whether results can be correlated very closely, as there may be a number of conflicting factors present. For instance, Smith and Quinn† show that the rate of absorption of CO_2 by alkaline carbonate solutions is influenced by the concentration of sulphate ions in solution to a degree that cannot be explained by ionisation changes. There are a large number of practical figures for the value of K_{G_a} available in literature, and it is better to choose figures for conditions as close to those planned as possible and to base design on these. Typical experimental values for K_{G_a} are set out in the table below.

Packing	Solute	Solvent	Gas	G	Kg_a	Reference
4-in. Partition Ring	NH_3	Water (450 lb./sq. ft./hr.).	Air	50 100 200	3 5 9	Kowalke, Hougen, and Watson, *Bull. Univ. Wiscon. Exptl. Sta.*, 1925.
Board Packing, 7-in × ¼-in. slats, 3¼-in. spacing.	Benzole	Gas oil (680 lb./sq. ft./hr.).	Coal gas	240	0·6	Sherwood, "Absorption and Extraction."
Board Packing, 4-in × ¼-in. slats, ⅞-in. spacing.	SO_2	NaOH solution (1000 lb./sq. ft./hr.).	Air	100 200 400	4·0 6·9 11·9	Sherwood, *loc. cit.*
1-in. Raschig Rings 1-in. Berl Saddles	CO_2	Na_2CO_3 solution (2000 lb./sq. ft./hr.).	Air	275	0·045	Furnas and Bellinger, *Trans. Amer. Inst. Chem. Eng.*, 1938, 34, 251 (Liq. Film Control).
Rotary Brush Washer.	NH_3	Water	Coal gas	1100	27	From Hollings and Silver, *Trans. Inst. Chem. Eng.*, 1934.
Spray Tower "Cyclone Scrubber."	SO_2	Na_2CO_3 in soln. (spray).	Air	900	1·5 —4·0	Johnstone and Kleinschmidt, *Trans. Amer. Inst. Chem. Eng.*, 1938, 34, 181.
Board Packing, 6-in. × ½-in. slats, ½-in. spacing.	Benzole	Creosote oil (200 lb./sq. ft./hr.)	Coal gas	130	0·4	Author's measurement on 8,000,000 cu. ft./day plant.
Raschig Rings, ½-in. × ½-in.	CO_2	NaOH soln. (1600 lb./sq. ft./hr.)	Air	190	1·3	Tepe and Dodge, *Trans. Amer. Inst. Chem. Eng.*, 1943, 39, 255.

Example 3. *The low-temperature carbonisation of coal is producing gas containing* 10 *lb. of light spirit*/1000 *cu. ft. This spirit has a mean molecular weight of* 80, *and the gas is being made at a rate of* 50,000 *cu. ft./hr. Design an absorption system for the recovery of* 90% *of this spirit by oil washing.*

* Hollings and Silver, *Trans. Inst. Chem. Eng.*, 1934, 12, 49.
† *Ind. Eng. Chem.*, 1941, 33, 1129.

The normal process of benzole recovery by oil washing is fully described in literature, and it is found possible to work up to 3% by weight of benzole in a wash oil of 0·9 sp. gr., while dealing with coal gas containing initially 2·5 lb./1000 cu. ft. The molecular weight of this gas benzole is approximately the same as the "light spirit" of the example. These final concentrations may be assumed to represent an experimental equilibrium, with Henry's Law holding for the range of the washing. As the final concentration in the solvent oil is 1·67 lb./cu. ft., the value of K will be 1·67/(2·5 × 10^{-3}), or 668. For a "slip", as in the example, of 0·1, the limiting value of Q will be*

$$\frac{1 - 0{\cdot}1}{668}, \text{ or } 1{\cdot}35 \times 10^{-3} \left(Q = \frac{1 - S}{K}\right)$$

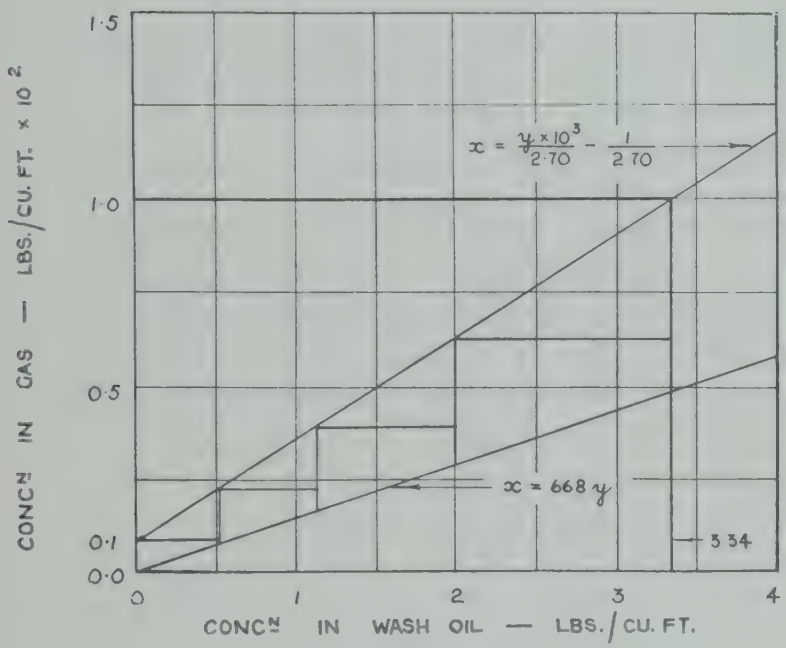


FIG. IV–15.

Making an arbitrary choice of 100% excess oil, Q becomes 2·70 × 10^{-3}, and the amount of wash oil in circulation will be 50,000 × 2·70 × 10^{-3}, or 135 cu. ft./hr. This will contain finally 450 lb. of light spirit or 3·34 lb./cu. ft. (5·9% by weight). If the solvent on entering the washer contains no light oil, the operation of the system can be represented by Fig. IV–15, and it will be seen that approximately four "70%" stages are required.

In view of the value of Q, a board-packed tower will be chosen.

* See, e.g., Hoffert and Claxton, "Motor Benzole".

Then a typical value for G, the gas bulk velocity, from the table above is 240 lb./sq. ft./hour ; this, with a gas of density about 0·05 lb./cu. ft., corresponds to a linear velocity of 1·33 ft./sec. The velocity fixes the tower cross-section, for 50,000 cu. ft./hour, at 10·5 sq. ft.—3·7 ft. diameter for a circular tower. The oil rate appears reasonable at 7600 lb./hour, or 720 lb./sq. ft.

Figures for K_{G_a} are quoted as 0·6 and 0·4 for board-packed towers, with higher figures for ring packings. In the circumstances a figure of 0·6 will be assured. In the first stage the concentration head varies from $(5{\cdot}0 \times 10^{-3})$ to $(1{\cdot}5 \times 10^{-3})$ lb./cu. ft.—a mean conctn. head of $2{\cdot}92 \times 10^{-3}$ lb./cu. ft. This for a mol. wt. of 80 is equivalent to a pressure of $1{\cdot}32 \times 10^{-2}$ atm., with 3·50 lb. of spirit being transferred per 1000 cu. ft. As 50,000 cu. ft. of gas passes per hr., this quantity is equivalent to 175 lb., or 2·2 lb. mols/hour, in all.

The volume V required for the first stage follows from the equation

$$2{\cdot}2 = 0{\cdot}6 \times 1{\cdot}32 \times 10^{-2} \times V$$

whence

$$V = 278 \text{ cu. ft.}$$

As the cross-section of the tower is fixed at 10·5 sq. ft., the height of the stage will be 26·5 ft. From symmetry considerations (Fig. IV–15) the other three stages will all be the same height, giving a total tower height of 106 ft. Such a tall, narrow tower would be built in two sections in series, each 53 ft. high (packed height) and 3·7 ft. in diameter. The number of "perfect theoretical stages" required is just under three, giving a height per stage of 35 ft.

The process as studied has dealt entirely with the absorption of a soluble component from gas mixtures by a suitable solvent. It is obvious that the process may be reversed, and a volatile material gradually stripped from a solvent mixture by a gas stream. If the same nomenclature is adopted as for gas absorption, then, referring to Fig. IV–8, a liquid stream has the concentration of volatile component reduced from x_0 to x_n, while concentration in the gas increases from $y_n(=0)$ to y_0. A mass balance, as before, shows that $y_0 = Q(x_0 - x_n)$ or for any stage $y = Q(x - x_n)$. This is a straight line on a normal operating diagram (Fig. IV–16), and cuts the X axis at x_n. With a slope Q it will lie below the equilibrium relation line $x = ky$. Then stepwise construction, starting from x_n, will give the number of theoretical stages required to pass from $y_n(=0)$ in the gas entering to y_0, the final concentration in the

enriched gas. The change to "70%" stages and determination of concentration head will follow as before. Few experimental data

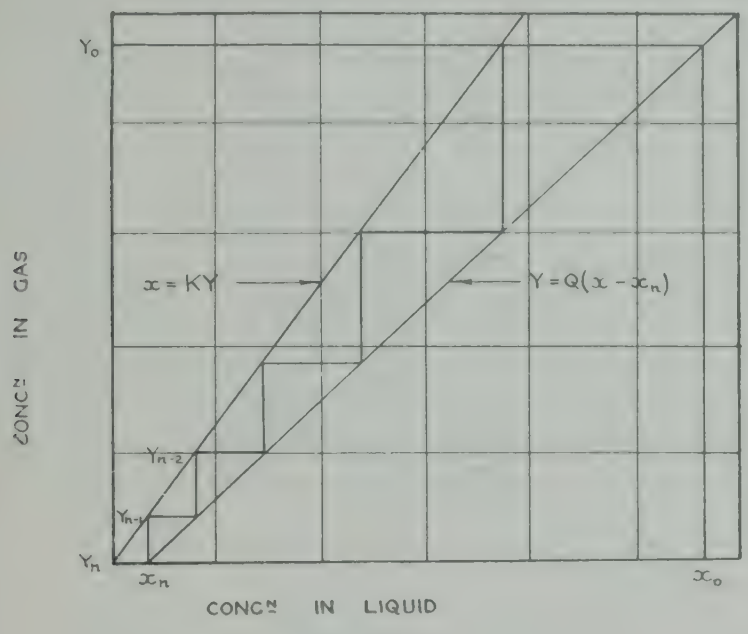


FIG. IV–16.

are available on rates of desorption, but it may logically be assumed that these will be of the same order as those observed in absorption.

CHAPTER V

EXTRACTION

The extraction of a soluble material from an insoluble residue, either liquid or solid, by means of a liquid solvent, has been variously termed extraction, leaching, or lixiviating. The two latter terms have been generally applied to removal of solute from solids, a process which has been very widely practised since the earliest years of technology. It is this treatment of solids which will be considered first, with a following section on liquid–liquid extraction.

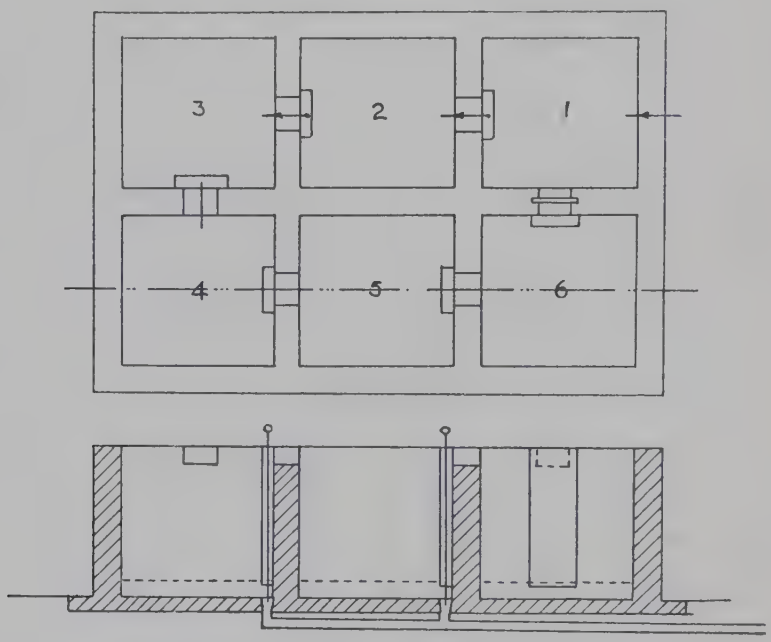


Fig. V–1.—Leaching-Tank Battery.

Long specialised experience in such processes as the preparation of wood extracts, of alkali from wood ashes, or of salts from mineral residues has led inevitably to the evolution of individual extraction plants. Certain general principles can be held as established, such as the use of a train of individual steeping vessels, in which extraction proceeds by stages, with fresh solvent for the partly extracted solid. The steeping vessels are often open-topped tanks with a false bottom

to support the solid and allow for the drainage of solution. In the tannin-leaching battery* of Fig. V–1, strong liquor from tank 1 is pumped or drained off after a suitable steeping period. Very weak liquor from tank 6 is then pumped to 5, and liquor of gradually increasing concentration overflows from tank to tank, ultimately refilling 1, in which the tannin bark will receive its second wash. The various tanks are now left undisturbed except for 6, from which the fully extracted bark is removed and a charge of fresh bark added. In the next step of the cycle, water is pumped to tank 5, displacing the extract from tank to tank as before, until the strong liquor (from tank 1) refills 6 to give the fresh bark its first treatment and to build up the liquor strength to the discharge value. This decantation system, with the solid remaining in one fixed position, is the first general method of counter-current extraction. The full cycle, for the system above, is set out in the following table.

Stage 1.	Pump strong liquor from 1. Pump 6 to 5, 5 to 4, 4 to 3, 3 to 2, 2 to 1. Empty 6 and refill.
Stage 2.	Pump water to 5, and hence 5 to 4, . . ., 1 to 6.
Stage 3.	Pump strong liquor from 6. Pump 5 to 4, . . ., 1 to 6. Empty 5 and refill.
Stage 4.	Pump water to 4, and hence 4 to 3, . . ., 1 to 6, 6 to 5.
Stage 5.	Pump strong liquor from 5. Pump 4 to 3, . . ., 6 to 5. Empty 4 and refill.
Stage 6.	Pump water to 3, and hence 3 to 2, . . ., 5 to 4.
Stage 7.	Pump strong liquor from 4. Pump 3 to 2, . . ., 5 to 4. Empty 3 and refill.
Stage 8.	Pump water to 2, and hence 2 to 1, . . ., 4 to 3.
Stage 9.	Pump strong liquor from 3. Pump 2 to 1, . . ., 4 to 3. Empty 2 and refill.
Stage 10.	Pump water to 1, and hence 1 to 6, . . ., 3 to 2.
Stage 11.	Pump strong liquor from 2. Pump 1 to 6, . . ., 3 to 2. Empty 1 and refill.
Stage 12.	Pump water to 6, and hence 6 to 5, . . ., 2 to 1.

This concludes the cycle, as Stage 13 is identical with Stage 1.

The gap between each operation stage will be a steeping time, in which the solvent comes into some degree of equilibrium with the solid. This steeping time may be long or short, according to the solid being extracted, and will be dealt with more fully below. In many cases the time for pumping and digging out is sufficient for steeping also, and the operations succeed each other without any check. The number of times that the bark is subjected to fresh solvent treatment is 11, and each fresh volume of water passes through 11 stages before final discharge. It can be shown that for

* M. B. Donald, *Trans. Inst. Chem. Eng.*, 1937, 15, 77.

N vessels in this process of "batch decantation" the number of treatments is $2N - 1$; that is to say, that the total extraction has comprised $(2N - 1)$ "equilibrium stages", in each of which the system solid–solute–solvent approaches equilibrium. The limiting factor in any such process will be the time taken for the extraction to reach a desirable degree of equilibrium. In the case of non-porous solids, associated only with such solution as cannot easily be drained out, this time can be very short, and, as mentioned above, be no more than the time required to complete such operations as pumping and discharge of spent solid.

When the solid being treated is in the form of porous vegetable matter, in which the solute proper is contained in microscopic cells, the extraction will necessarily be much slower. Normally these solids are sliced or crushed to present the greatest possible surface to the extracting fluid. Typical instances of this are found in the multi-roll cane-sugar mill, or in the slicing of sugar-beets into "cosettes" or small slices of irregular form. This makes for complexity in the following extraction process, as the solid surfaces present either a cell wall—of a varying thickness—or a broken cell to the solvent. Solute from the broken cell is easily removed, but diffusion of solvent into and subsequent diffusion of solute out of unbroken cells will take very much longer. It is generally impossible to continue the disintegrating process until all the vegetable cells are broken open, as these are very small and the tiny vegetable fragments would form with the solution an almost unfilterable mass. In these circumstances it is obviously impossible to give any general rule for the time taken to reach equilibrium in an extraction stage, and even with one specific type of vegetable matter it is not likely that rates of extraction will follow any rigid mathematical form.*

It is probably true that any extraction can be regarded as being movement of the solute across two resistances in series, the first being that offered by the porous solid material, and the second that of the stagnant film of liquid at the interface. From a general consideration of liquid-film principles, it is clear that the second resistance could be diminished by circulating the liquid rapidly through the solid, and that diffusion rates will increase as the viscosity of the liquid decreases. Plants have been devised in which the liquid was circulated through a calorifier and considerable increases in

* Fan and Morris, *Ind. Eng. Chem.*, 1945, 40, 195.

extraction rate obtained.* At the same time, it is possible that high temperatures and rapid circulation may produce unfilterable slimes, so these improvements are not always applicable.†

The flat-bottom tank battery does not lend itself to rapid discharge, and the rectangular form shown is unsuitable for fabrication in metal, or for withstanding either pressure or vacuum. These operating conditions must be met, on a very large scale, in the

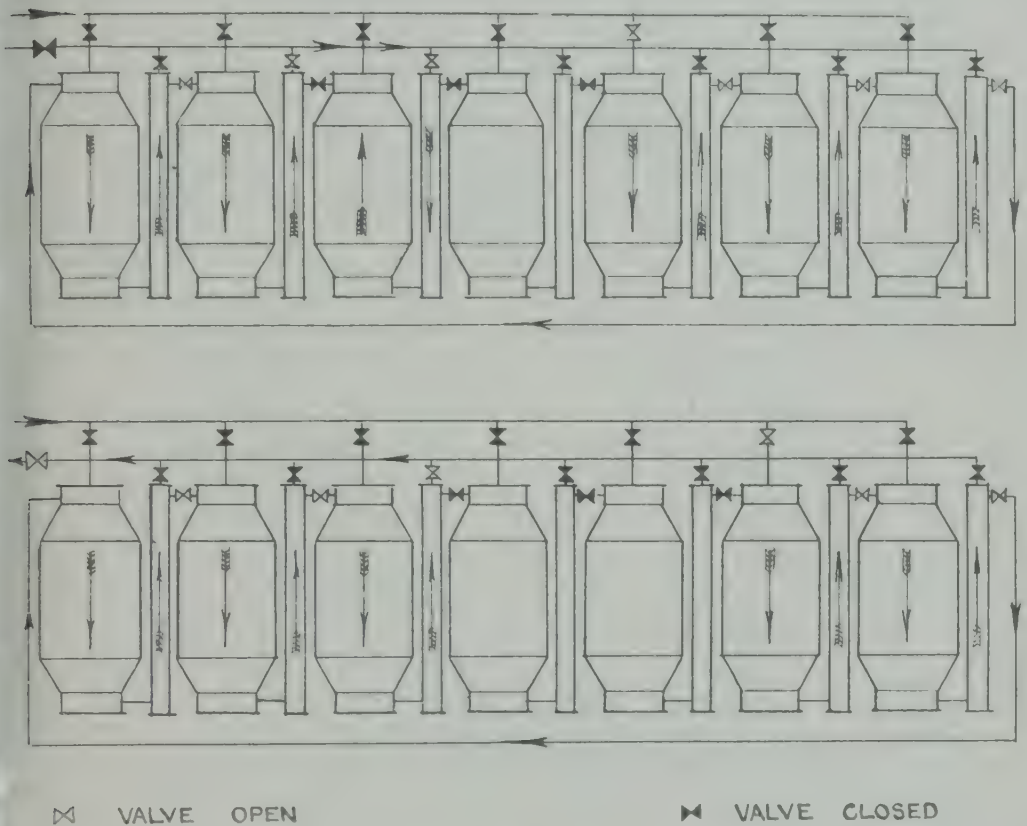


Fig. V–2.—Sugar-beet Diffusion Battery.

extraction of sugar-beet. The beet extraction unit, or diffuser, is a cylindrical vessel with a conical base, ending in a large discharge valve. The layout of a diffusion battery is shown in Fig. V-2 and a typical diffuser in Fig. V–3. The beet is cut into slices from 0·25 to 0·5 in. in thickness, and in this form can be charged from an overhead hopper or washed out through the bottom discharge. A very large number of patent devices have been introduced into these batteries to facilitate extraction or handling.

* Peck, *Quart. J. Pharm.*, 1936, 9, 401.
† Donald, *loc. cit.*

A battery of diffusion tanks is essentially a plant for batch operation, and as such does not lend itself to labour economy and automatic control as readily as does a continuous process. The structure of the beet-sugar industry is such that much labour is engaged on a short seasonal basis, and it would be very desirable if a more labour-saving form of extraction could be devised. Nevertheless, the bulk of the world's beet-sugar factories still work on these batch diffusers. This is because any conveying device breaks up the fragile vegetable

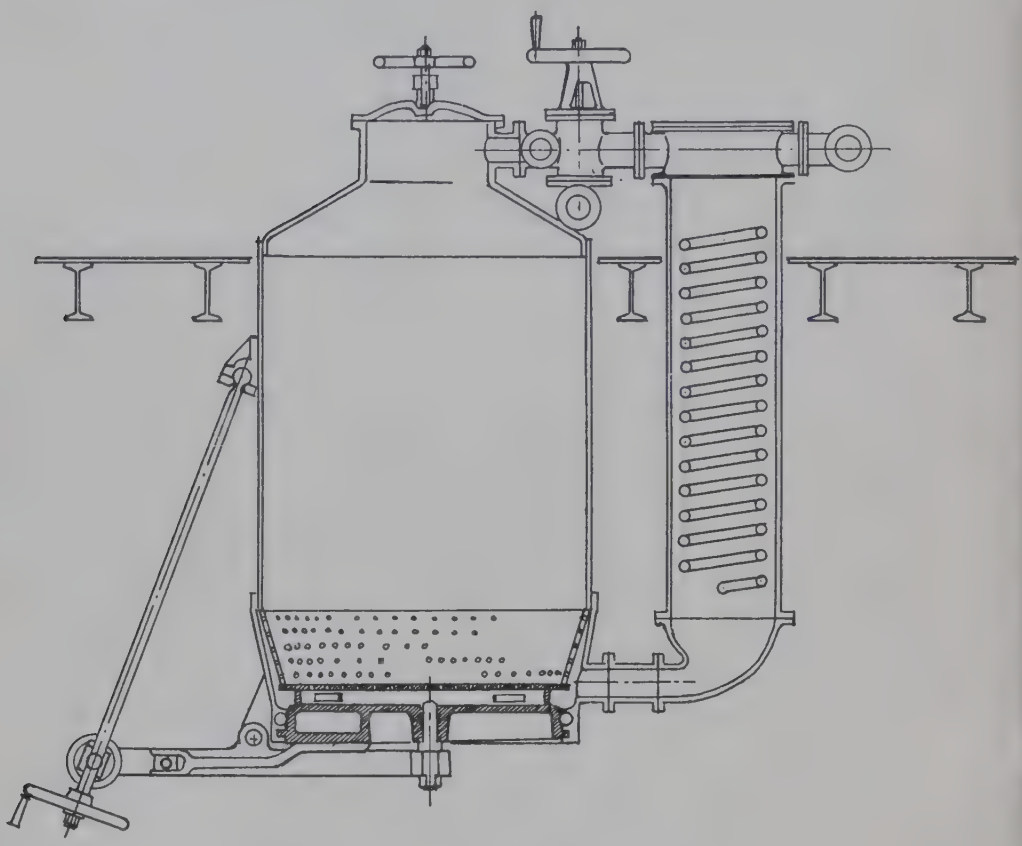


Fig. V–3.—Beet Diffuser.

matter and produces an unfilterable but reactive slime. Conveyor systems have been operated and a typical design is shown in Fig. V–4. In these both liquid and solid phases move, and no definite "stage" exists; the change is almost the same as with distillation, in transfer from a plate column to a packed column. In extraction, however, the number of theoretical stages may be modified as the second phase starts to move, and it is desirable at this point to go more fully into the underlying principles of extraction processes.

Consider a system of N vessels or stages through which flows a

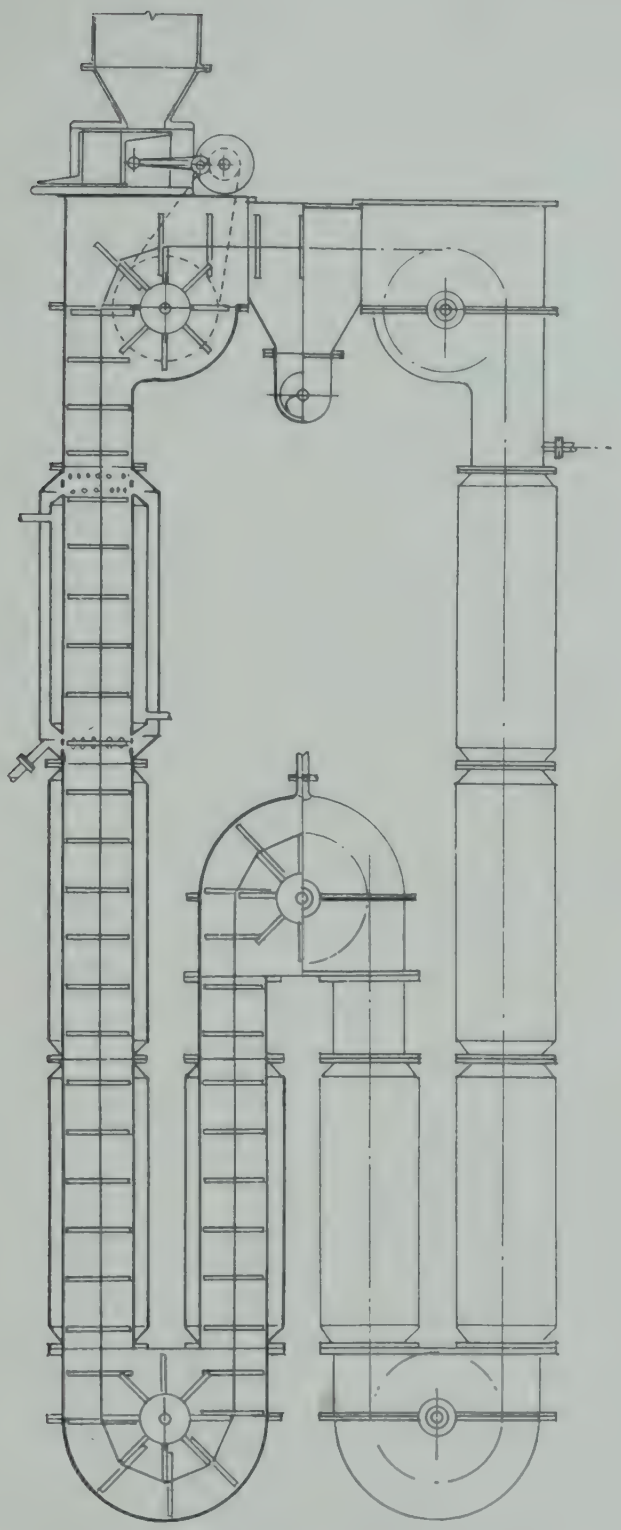


FIG. V–4.—CONTINUOUS BEET EXTRACTOR.

solvent stream containing S lb. of pure solvent, and a solid material for extraction containing W lb. of dry inert solid. It may be assumed initially that the ratio of W to S is constant for each stage; that is to say, that the solid enters the Nth stage carrying with it a definite weight of solvent, and this passes unchanged from stage to stage, leaving finally with the extracted solid from the first stage, at which the pure solvent enters. Fig. V–5 represents the flow of materials in the process. In the diagram, concentrations of solute, in lb./lb. of solvent, are shown as x_1, x_2, . . ., with suffixes to indicate the stage the stream is *leaving*, and concentrations of solute in the solid, in lb./lb. of dry inert solid, are shown as y_1, y_2, . . ., with suffixes to indicate the stage that the solid stream is *entering*.

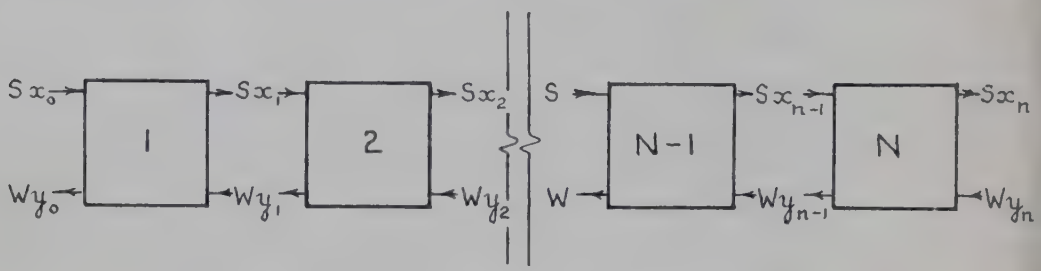


Fig. V–5.

A mass balance over the whole process gives

$$S(x_n - x_0) = W(y_n - y_0)$$

or, if $x_0 = 0$,

$$Sx_n = W(y_n - y_0)$$

If the ratio S/W be denoted by Q, then the equation becomes

$$x_n = \frac{W}{S}(y_n - y_0), \text{ or } x_n = \frac{y_n}{Q} - \frac{y_0}{Q} \quad . \quad . \quad . \quad (1)$$

In an "ideal" extraction the solute dissolves in the solvent completely, and a certain proportion of the resulting solution passes in one direction with the solid stream, while the residue forms the solvent stream. When the amount of solvent associated with the solid stream is constant, as assumed above, the ratio R of solute leaving any stage in the solution stream to solute leaving in the solid stream is constant.

Therefore, for any stage, M in number,

$$RWy_{m-1} = Sx_m, \text{ or } x_m = \frac{R}{Q}y_{m-1} \quad . \quad . \quad . \quad (2)$$

The relationships (1) and (2) can now be drawn on an X, Y diagram as in Fig. V–6, with concentrations in the solvent stream on the X axis, and those in the solid stream on the Y axis. In the first stage pure solvent enters ($x_0 = 0$) and dissolves out solute to reach a concentration x_1 such that $x_1 = R_1 Q \cdot y_0$. A mass balance over this first stage shows that

$$Sx_1 = W(y_1 - y_0), \text{ or } x_1 = \frac{y_1}{Q} - \frac{y_0}{Q}$$

giving a value for y_1. From this value of y_1, $x_2 = R_1 Q \cdot y_1$ and so on. But this is the usual stepwise construction from line to line

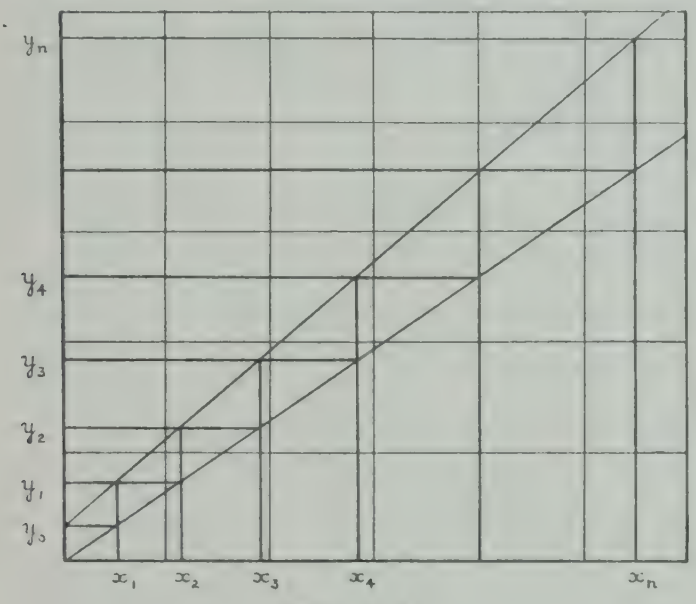


FIG. V-6.

of the diagram, giving the number of stages required to pass from y_0 to y_n—five stages in the diagram, terminating at $y_5 = y_n$. It may be supposed, however, that each of these steps represents complete equilibrium between liquid and solid, which in turn demands mixing at each stage for a considerable time. In fact, if equilibrium is approached by some normal logarithmic relation, the time required for perfect equilibrium will be infinite. If a definite time is chosen which gives a 70% approach to equilibrium, then the changes will be as set out in Fig. V–7, where the vertical at $x_{\bar{1}}$ is drawn so that $Ox_{\bar{1}} = 0{\cdot}7\, Ox_1$. A balance over the first stage shows that $Sx_{\bar{1}} = W(y_1 - y_0)$, or the intersection of $x = x_{\bar{1}}$ with the upper operating line gives y_1 as before. A series of "70%" ideal stages will pass from y_0 to y_n with a definite time in each stage.

It has been supposed that the solid enters the system " wet " with solvent. This may not be the case, and the solid can enter the n^{th} stage dry. It will then entrain a certain amount of solvent of weight α, whence the weight of solvent leaving the n^{th} stage becomes $S - \alpha$. By a mass balance over this stage

$$(S - \alpha)x_n = W(y_n - y_0), \text{ or } x_n = \frac{W}{S - \alpha}(y_n - y_0)$$

It will again be supposed that equilibrium is reached in each stage, when the solution carried forward by the solid has the same concentration of solute relative to solvent as the solvent stream flowing in

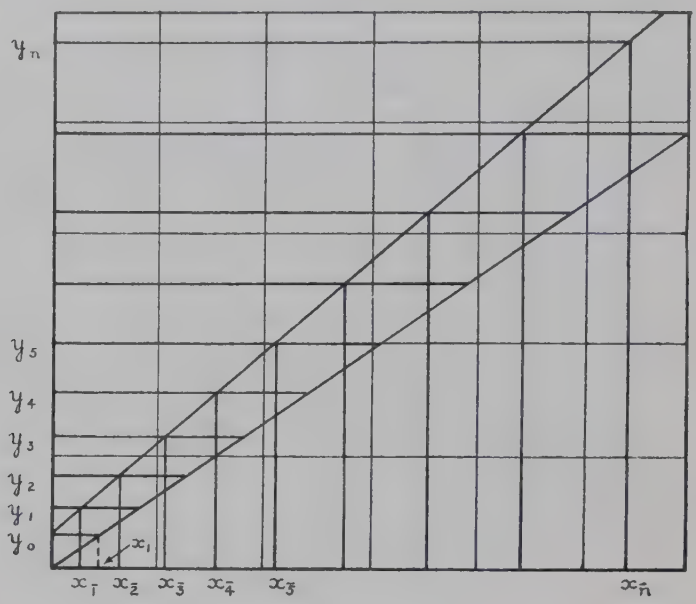


FIG. V–7.

the other direction. Then in all stages of the extraction up to the n^{th} stage, the weight of solute in the solid stream is αx and the weight in the solvent stream is Sx. But these quantities have a fixed ratio R, from which

$$R\alpha x = Sx, \text{ or } \alpha = \frac{S}{R}$$

In the final (n^{th}) stage, the amount of solute in the solid stream leaving must be αx_n, whence $Wy_{n-1} = \alpha x_n$.

$$\therefore\ x_n = \frac{W}{\alpha}y_{n-1} = \frac{WR}{S}y_{n-1} = \frac{R}{Q}y_{n-1}$$

The relation between the streams leaving the final stage is therefore still represented by the general equation $x = R\,Q\,.\,y$, but the relation between solid stream entering and solvent stream leaving has altered. Graphical construction for this system is shown in Fig. V-8, where the stepwise movement between the operating lines stops at the value x_n given by

$$x_n = \frac{W}{S - \alpha}(y_n - y_0)$$

So far it has been supposed that a regular relation exists between concentration in the various stages. This is analogous with the

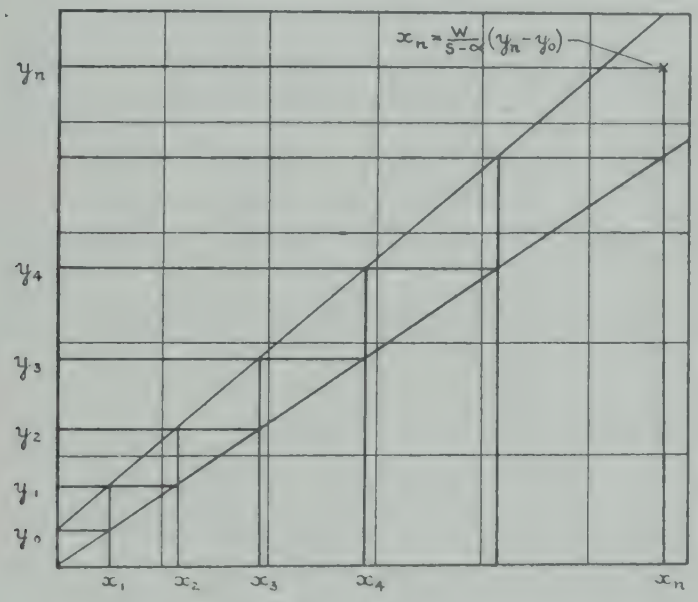


FIG. V-8.

supposition that the relation between vapour and liquid compositions in distillation follows a regular form. As with distillation problems, a quite irregular relationship may be expressed graphically in the same way for mixtures of liquid solvent and solid. If a series of stages of extraction are considered, as in Fig. V-9, where the weight of both solvent and solid streams varies, then for any stage M the relation between the two streams leaving will be $S_m x_m = K_m W_{m-1} y_{m-1}$ and the relation between solvent stream leaving and solid stream entering is, on a mass balance, $S_m x_m = W_m y_m - W_0 y_0$. These corresponding values can be determined experimentally by stirring solvent with solid for a sufficient time for equilibrium to be almost established, separating the phases,

and analysing for solvent, solute, and solid. When a sufficient range of values has been obtained they can be plotted on two curves as shown in Fig. V–10, and will normally tend to assume some reasonably smooth curve. The stepwise construction from pure

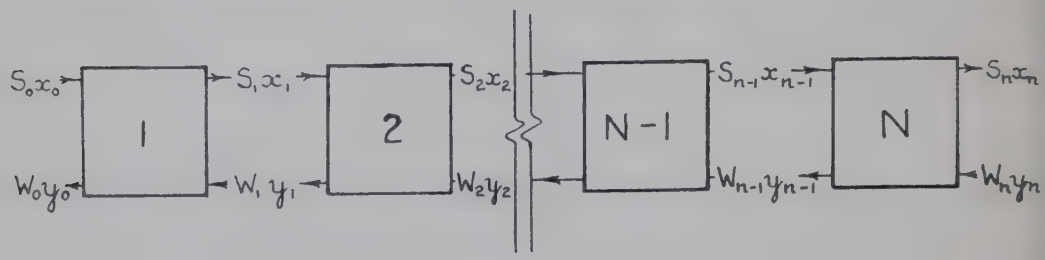


Fig. V–9.

solvent entering remains the same, while "70%" approaches to equilibrium can also be assumed. The construction terminates on the co-ordinates x_n, y_n where $x_n = \dfrac{W_n y_n - W_0 y_0}{S_n}$.

If these graphical constructions are to be translated into practical

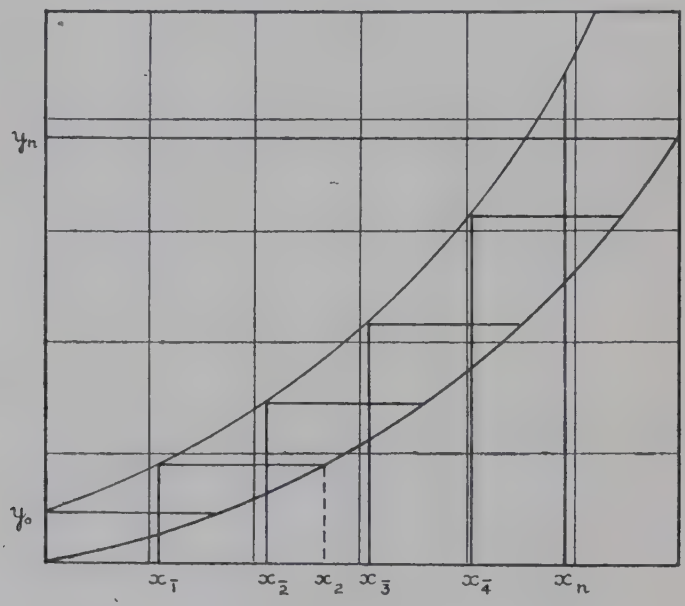


Fig. V–10.

plant design, it will be necessary to determine the time required for a definite approach to equilibrium. It has already been suggested that the driving force in extraction may be analogous to the concentration head of gas absorption practice. If the equilibrium concentration in the solvent is C_e, and the actual concentration C at

any time T, then the rate of extraction on this basis will be given by $dC/dT = K(C_e - C)$. Following the normal processes of integration, it can be shown that the time T taken to reach a concentration C from an initial concentration C_0 when the equilibrium figure is C_e will be $\frac{1}{K}\log_e \frac{C_e - C_0}{C_e - C}$. Here K is some experimental constant. Attempts to verify this equation experimentally have shown that a number of factors complicate the simple relationship. The effect of the various forms of cell in sliced vegetable matter have been noted by Osborn and Katz,* and later by Fan and Morris.† The most practical effort so far appears to be by Ruth,‡ who, arguing from the work cited above, suggests a practical expression (for soya-bean flakes) of

$$T \text{ (min.)} = K\left(\frac{C - C_0}{C_e - C}\right)^{0.918}, \text{ where } K = 0{\cdot}068\ (10^{79{\cdot}5\theta}).$$

Here θ is the flake thickness in inches, and C_e, C_0, C, concentrations in lb./cu. ft. of solution of the oil being extracted. Now the ratios of concentrations in lb./cu. ft. will not be markedly different, in dilute solutions, from the ratios of lb./lb. of solvent, as used above. In a stepwise construction, as in Fig. V–10, the length $x_{\overline{1}}$–$x_{\overline{2}}$ represents $C - C_0$, and the length $x_{\overline{2}}$–x_2 represents $C_e - C$. The ratio of these lengths will clearly be constant, for all "70%" stages, at $\frac{7}{3}$ or 2·3. In a typical case, where the thickness of the nut flakes is 0·025 in., $K = 0{\cdot}068\ (10^{1{\cdot}99})$, or approximately 6·8. Then the time for each stage will be $6{\cdot}8\ (2{\cdot}3)^{0{\cdot}918}$, or 14·6 min. These figures can be used as a basis for plant design.

Example 1. *Nut flakes of* 0·025 *in. in thickness, containing* 16% *of oil, are to be extracted to leave a residue containing on a dry meal basis,* 0·75% *of oil. The finished extract is to contain* 20% *by weight of oil in a light petroleum solvent, while the wet meal will contain* 50 *lb. of solvent per* 100 *lb. of dry meal. Calculate the number of extraction stages required.*

The oil content per lb. of inert meal is initially 0·1906 *lb. and finally* 0·0076 *lb. In the final solution* 0·25 *lb. of oil are associated with* 1 *lb. of solvent.*

Using the nomenclature adopted above, and assuming that the nut

* *Trans. Amer. Inst. Chem. Eng.*, 1944, 40, 511.
† *Loc. cit.*, p. 126.
‡ *Chem. Eng. Prog.*, 1948, 44, 71.

flakes are initially solvent-free, but are associated with a constant weight of solvent after the N^{th} stage, a mass balance gives

$$0{\cdot}25(S - \alpha) = W(0{\cdot}1906 - 0{\cdot}0076)$$

$$\{x_n(S - \alpha) = W(y_n - y_0)\}$$

But $\alpha = 0{\cdot}5W$, *and therefore* $0{\cdot}25S - 0{\cdot}125W = 0{\cdot}183W$ *or* $0{\cdot}25S = 0{\cdot}308W$, *whence* $\dfrac{S}{W} = \dfrac{0{\cdot}308}{0{\cdot}250} = 1{\cdot}23 = Q$.

It follows that $\alpha = \dfrac{0{\cdot}5}{1{\cdot}23}S$. *Assuming that the concentration*

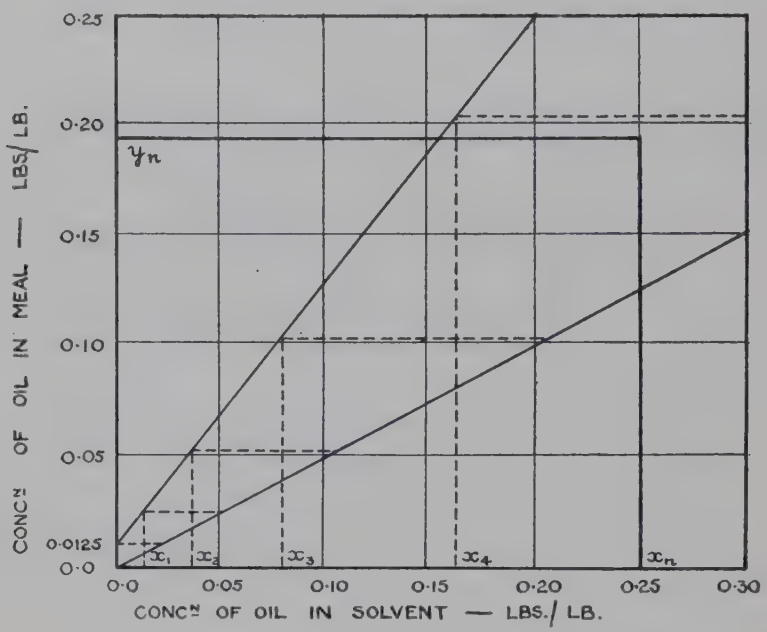


FIG. V–11.

of the solution leaving in the solid stream is equal to the concentration in the solvent stream, the ratio $R = \dfrac{S}{\alpha} = \dfrac{1{\cdot}23}{0{\cdot}5} = 2{\cdot}46$. *Then the two operating lines become*

$$x = \frac{2{\cdot}46}{1{\cdot}23}y \text{ and } x = \frac{y}{1{\cdot}23} - \frac{0{\cdot}0076}{1{\cdot}23}$$

and are drawn in Fig. V–11. Stepwise construction from $y = 0{\cdot}0076$ *up to* $y = 0{\cdot}1906$, $x = 0{\cdot}25$ *shows that between four and five "* 70% *" stages are required. For a safe working margin five stages will be specified.*

Then the time required for each stage will be 14·6 min., giving a total contact time of 73 min. If the process is to be one of counter-current decantation, then three vessels will be required to give five stages. These vessels are filled with meal and solvent. If the five stages are each to take 15 min., then the total capacity of the three vessels must be equal to the volumes of flake and meal handled over 75 min. Consideration of the batch size will suggest a suitable design.

Reverting to the original scheme of extraction as set out in Figs. V–5 or 9, it will be realised that if both solvent and solid are actually moved from one vessel to another in the process, the number of vessels required to give N stages will also be N. In the more general form of this arrangement, where the solid is moved steadily by some type of conveyor, through the solvent stream, it may be supposed that a certain length of the plant will be equivalent to an equilibrium stage, or to a "70% " stage. In the example above, if a velocity of 0·5 ft./min. in either direction can be obtained and the formula for extraction rate still holds, then the length of the conveyor would need to be 37·5 ft., and the total volume would be sufficient to contain all the material handled in 75 min., as before. Unfortunately for the full testing out of this scheme of design, practically no working figures for the rate of attainment of equilibrium are available. In the normal working of beet-sugar batteries it would appear that little attempt is made to approach equilibrium. With a typical battery of 12 diffusers the time of steeping is determined by the speed with which cells may be discharged and refilled, and it is probable that a stage efficiency of about 50% is the most that is attained. In a note on the extraction of copper ores with ammonia solution W. H. Dennis* suggests that a 12-hr. steeping period will give a 75% efficiency. It is probable that the process will always vary widely in approach to various types of solid.

Liquid–Liquid Extraction. This process can only be carried out when the solvent used for extraction is immiscible, or nearly immiscible, with the original liquid–solute residue. Now any system of three components can be represented by a triangular diagram, as in Fig. V–12, in which the three vertices represent the pure components. For extraction it is necessary that the greater part of the area within this triangle shall represent conditions in which

* *Ind. Chemist.*, 1942, 18, 427.

ternary mixtures will separate into two phases of an approximately binary character. Thus, in Fig. V 12, if a solution of solute B in residue W is to be extracted with solvent S, all mixtures of the three components represented by points in the area $y_a P x_a$ will separate into two phases. In a typical case these may be represented by y_a, x_a. The ternary mixture which has separated into these two phases will be represented by the point M on the line joining y_a, x_a. The line y_a–M–x_a is known as a "tie-line" and can only be determined experimentally.

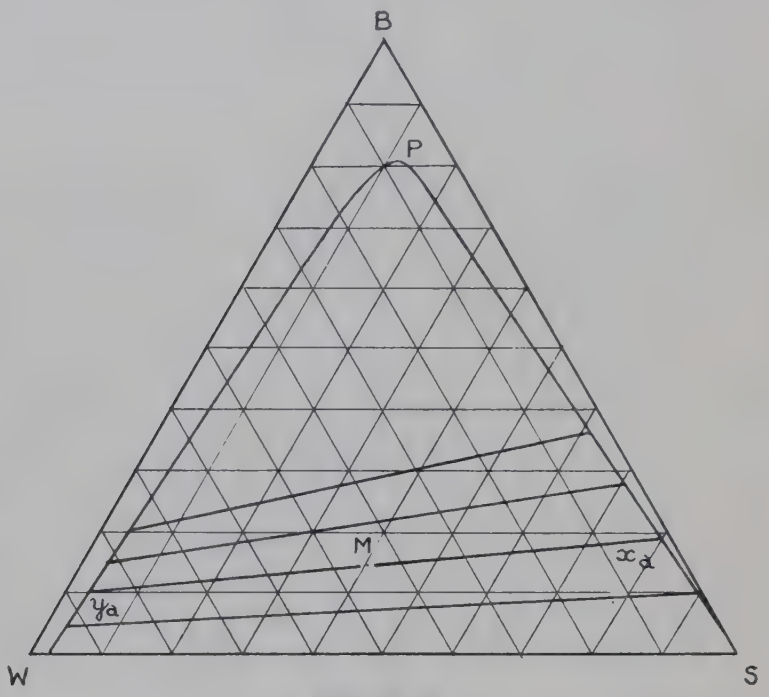


FIG. V–12.

Now the process of extraction, carried out in some type of counter-current plant, will consist in separating the solvent-rich x_a from the residue y_a and bringing these two into contact with other mixtures. The whole process can again be represented by a diagram such as Fig. V–9. Here S_1, S_2 represent the weight of solvent in the solvent stream leaving appropriate stages, while x_1, x_2 are the weights of solute B associated with unit weight of solvent. Also W_1, W_2 and y_1, y_2 are corresponding weights of residue and of solute associated with the residue.

Then, as before, for any stage,

$$S_n x_n = W_n y_n - W_0 y_0 \text{ and } S_n x_n = R_n (W_{n-1} y_{n-1})$$

These two equations give the operating lines for a graphical representation of extraction. In general, however, values of S and of W will vary widely from stage to stage, and it may not be possible to construct the diagram without a great amount of experimental work. If component W is almost insoluble in component S, as shown in Fig. V-12, the values of W and S will remain almost constant from stage to stage for concentrations of B below about 50%, and it is possible to make calculations on the same basis as was used for solid leaching. The slope of the tie-lines will give the relative concentrations of solute in residue and solvent streams which come to equilibrium in each stage, and these can be read off from the diagram of Fig. V–12.

Example 1. *A soluble component B is being extracted in counter-current flow by a solvent S from a residue W, the three-component system being represented by Fig. V-12. The initial concentration of B in W is 25% by weight, and the weight of solvent used is equal to the initial weight of solution. How many stages will be required to reduce the concentration of B in the residue down to 2%?*

On a weight basis the original content of solute B is 0·33 lb./lb. of W, and this is reduced to 0·02 lb./lb. by a solvent S of weight 1·33 W.

An approximate mass balance gives

$$S_n x_n = W(0{\cdot}33 - 0{\cdot}02), \text{ or } 1{\cdot}33 W x_n = W(0{\cdot}31)$$

whence $x_n = 0{\cdot}233$. The first line of the operating diagram will be given by $1{\cdot}33x = y - 0{\cdot}02$—assuming W and S constant. In any stage $1{\cdot}33 W x_m = RW y_{m-1}$, where x_m and y_{m-1} are complementary compositions at either end of a tie-line in Fig. V–12. The following values for concentration of solute can be read from the diagram:

$y_a =$	0,	0·05,	0·10,	0·15
$x_a =$	0,	0·10,	0·19,	0·26

Or, in lb./lb. of residue and solvent, and in the terms above:

$y_{m-1} =$	0,	0·053,	0·111,	0·177
$x_m =$	0,	0·011,	0·222,	0·352

This gives the locus of the second operating line, as shown in Fig. V–13. Stepwise construction shows that just over three "70%" equilibrium stages are required.

When the three components are mutually soluble to some extent, as in the system depicted in Fig. V-14, then calculation of the actual

weights passing from stage to stage will become complicated, and it will be desirable to use another system of graphical computation, that introduced by Hunter and Nash.* In the multi-stage process

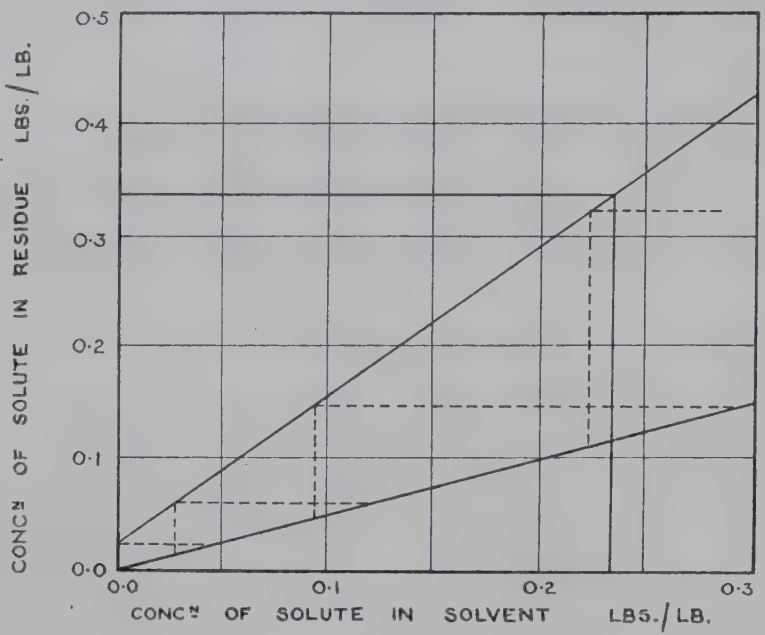


FIG. V–13.

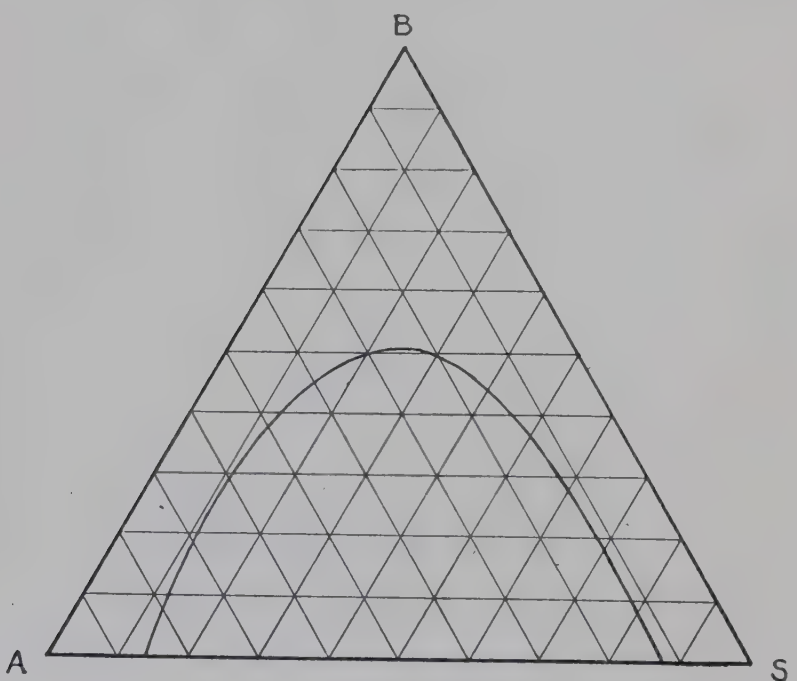


FIG. V–14.

* *J.S.C.I.*, 1934, 53, 95T.

represented in Fig. V–15, let H_m be the total weight of solvent stream leaving the M^{th} stage, and L_m the total weight of solution stream entering. The three-component system will be a mixture of A and B, originally represented by D in Fig. V–16, and this is to be

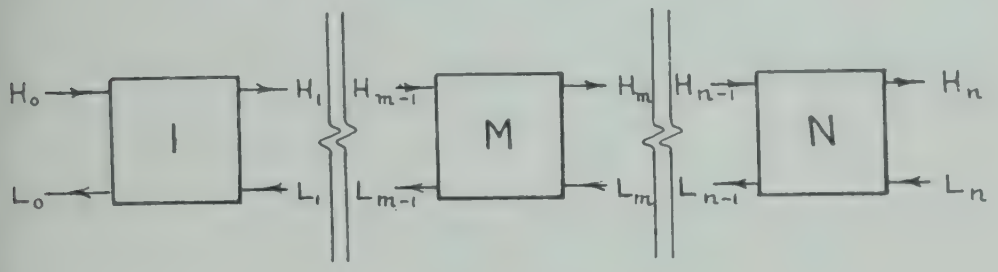


Fig. V–15.

extracted with solvent S to give a final residue of composition represented by J. By a mass balance over the M^{th} stage

$$H_{m-1} + L_m = H_m + L_{m-1}, \text{ or } H_{m-1} - L_{m-1} = H_m - L_m$$

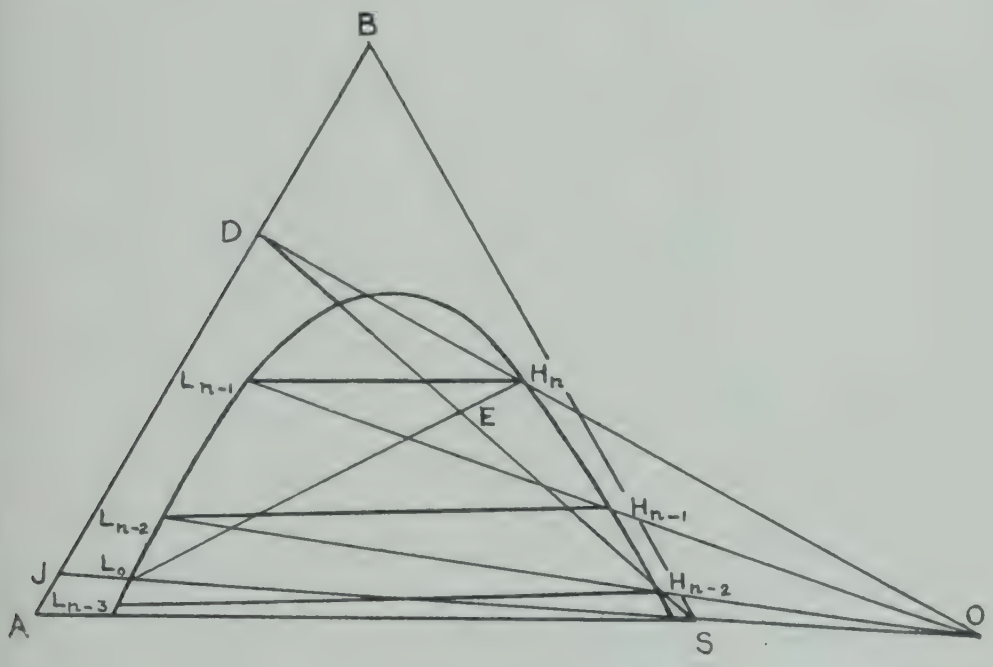


Fig. V–16.

Similarly, for the $M + 1$ stage,

$$H_m - L_m = H_{m+1} - L_{m+1}$$

and so on, or the weight of the mass stream passing from left to right in Fig. V–15 is constant.

If the concentration of any component A in the solvent stream leaving the M^{th} stage is a_m, while that in the solution stream entering is $\bar{a}_m$, then a mass balance for this component shows that

$$H_{m-1}a_{m-1} - L_{m-1}\bar{a}_{m-1} = H_m a_m - L_m \bar{a}_m$$
$$= H_{m+1}a_{m+1} - L_{m+1}\bar{a}_{m+1} \cdots$$

The mass stream passing from left to right will therefore have a constant composition as well as a constant weight. It may therefore be represented by some point O in a co-ordinate diagram for A, B, and S. This point need not lie inside the triangle of real compositions, but may have a negative amount of one or more components and lie outside. The composition of the solution leaving the final stage will be determined by mixing pure solvent, of composition represented by S, with a mixture of A and B in the proportions represented by J. It is a fundamental property of co-ordinate diagrams that the composition of a complex formed by combining two mixtures will lie on the line joining the two points representing these two components. Then the point representing L_0 (Fig. V–15) must lie on the line SJ. As this mixture has separated from a solution-rich phase, it must lie on the equilibrium curve for such mixtures, at L_0.

Now over the whole extraction process $L_n + H_0 = L_0 + H_n$, or the complex formed by mixing the inlet streams will be equal to the mixture of outlet streams. As S represents the initial solvent composition H_0, and D the initial solution composition L_n, then the complex composition $H_0 + L_n$ must lie on SD. The actual position is located by the relative weights of the compounding mixtures and will lie at E, so that $SE : ED :: L_n : H_0$. But $L_0 + H_n$ will also combine to give a mixture E, so that H_n will lie on L_0E produced to cut the equilibrium curve at H_n. Now it has been shown that $H_0 - L_0 = H_n - L_n$, or, substituting, $S - L_0 = H_n - D$. The point O must lie on the lines L_0S, DH_n, and can be located by producing these lines to their intersection, as shown in Fig. V–16. In the final or N^{th} stage, H_n will be in equilibrium with L_{n-1}, the solution stream leaving this stage. The point L_{n-1} will lie at the end of the experimental tie-line from H_n. As $H_{n-1} - L_{n-1} = 0$, the line joining L_{n-1} to O will cut the equilibrium curve at H_{n-1}. Then a tie-line from H_{n-1} will determine L_{n-2}, and the construction proceeds until a tie-line passes through or past L_0. In the diagram this occurs at the third stage, or between two and three stages will be required for the extraction.

Example 2. *A mixture of aromatic hydrocarbons (B) in a paraffin solution (A) is to be extracted by a preferential solvent S, so that an initial aromatic content of 60% by weight is reduced to 8%. The properties of the ternary mixture are represented by the triangular diagram and equilibrium curve of Fig. V–16, and 80% by weight of solvent (on original solution weight) is to be used for the extraction. How many stages will be required, and what will be the final compositions of solution and solvent extract?*

The construction for this problem is shown in Fig. V–16. The point E is located on SD by the fact that SE : ED : : 100 : 80, and the compositions of H_n and L_0 can be read off directly from the diagram. They are respectively:

	Residue (L_0)	*Extract* (H_n)
Solvent (*S*) . .	9%	52%
Aromatic (*B*) . .	7%	41%
Paraffin (*A*) . .	84%	7%

The extraction will have to be carried out in three stages.

This method has the advantage that the limitations of the field in which extraction can be carried out are immediately obvious, but it is often difficult to draw accurately a diagram in which triangular co-ordinates are used and the construction limited largely to the lowest sections of the triangle. This is not infrequent in cases of solvent extraction, and the difficulty may be overcome by the suggestion of Kinney* that the equilateral triangle of co-ordinates be replaced by a right-angled triangle diagram.

No very special plant for liquid–liquid extraction has been developed. The ordinary packed tower, or even bubble-cap tower, can be used in the same way as for gas washing, with the lighter liquid entering at the bottom and flowing upwards in a tortuous path through the heavier liquid descending. Either phase may be dispersed in the other, and no general rule for rate of mass transfer has been propounded. If droplets of one fluid are suspended in another, then the velocity of the droplets should be as high as possible if the film resistance outside the droplet is to be small. It has been suggested,† therefore, that the liquid present in greatest volume should be dispersed in the other – a state of affairs which may be difficult to obtain. In addition, too large an upward velocity of the lighter phase, or *vice versa*, may lead to the stoppage of liquid

* *Ind. Eng. Chem.*, 1942, 34, 1102.

† *Trans. Amer. Inst. Chem. Eng.*, 1942, 38, 179.

flow in the opposite direction, the column being said to " flood ". In view of the relatively small differences between liquid densities which are responsible for the proper working of such plant it would appear logical to use a centrifugal washer, built in zones, to carry out a number of extractions in series, with light liquid flowing inward and heavy liquid outward. Such a machine has been patented,* but working details are not available.

To overcome the difficulty of rapid movement for contact, followed by the need for complete separation, plants have been built in the form of a series of mixing vessels coupled to an equal number of separators. The first of these was originally devised for a continuous nitration reaction, in which hydrocarbon and acid followed opposite directions through the plant, but was afterwards put into use for liquid–liquid contact under the name of the " Holley-Mott " washer. The general principles of the plant are shown in the diagram of Fig. V–17. The vessels M are the mixers, provided with stirring devices, and these are connected at top, bottom, and middle to the separators S. If the two liquids have respective densities ρ_1 and ρ_2, then the mean density in the mixer will be $\frac{\rho_1 + \rho_2}{2}$. At the middle of the separator a head H of density ρ_1 will be balancing an equal head of density $\left(\frac{\rho_1 + \rho_2}{2}\right)$. As these will be out of balance, flow of the mixture along the middle connection from mixer to separator and flow of light liquid back to the mixer will go on steadily. Similarly at the lower connection a flow of heavy liquid from separator to mixer will be set up whenever the stirrer is in motion, with flow from mixer to separator along the middle connection as before. If a light liquid is fed in at one end of the plant, it will flow steadily from separator to separator and out at the far end, by normal hydrostatic head. A similar flow of heavy liquid in the opposite direction can be effected, without any appreciable difference in liquid level from vessel to vessel, and an overall hydrostatic head of about 1 in. in either direction for 16 pairs of mixer-separator units in a typical washing operation. A somewhat similar principle has lately been devised† for a tower washer, in which a series of mixing zones are spaced between separating zones of static packing.

* U.S.P. 2, 291, 849.

† Scheibel, *Chem. Eng. Prog.*, 1948, 44, 681.

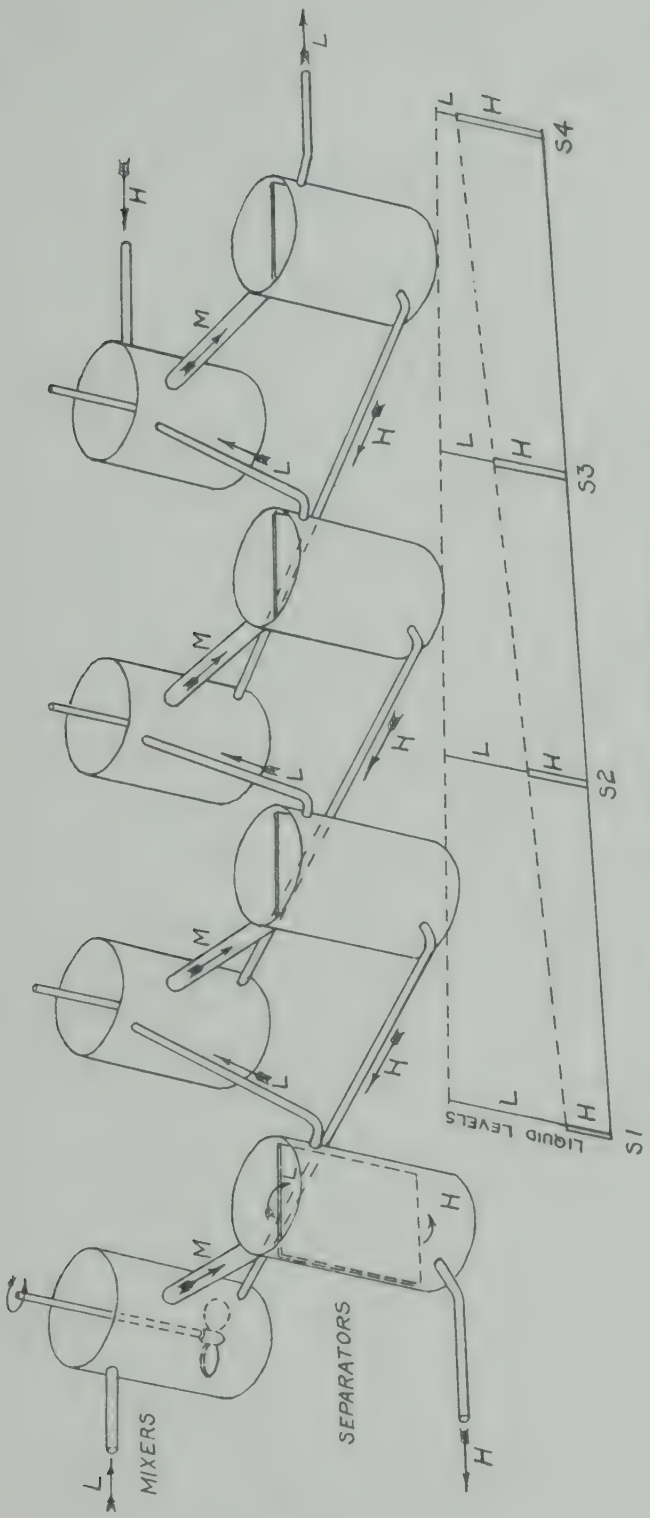


FIG. V-17.—HOLLEY-MOTT WASHER.

A process which has been developed in the laboratory for the separation of complicated chemical mixtures has made use of the differential absorption of the components on some activated solid surface. This is equivalent to counter-current extraction, with the solid column taking the place of the extracting liquid. Up to now no attempts have been made to move the solid, but this may be a development of the immediate future. Large " chromatographic " units have been built, however, and the slow rate of flow of liquids through finely divided porous solids has been accelerated by centrifugal force.*

* B.P. 585, 224.

CHAPTER VI

EVAPORATION

This process may be defined as the removal of a volatile material from solutions containing initially only small quantities of a solute of negligible vapour pressure. The problem is essentially one of heat supply and rapid removal of the vapours formed. The heat may be supplied directly, by hot gases or radiant heat coming into direct contact with the liquid, or indirectly through a metal wall: the vapours can be swept away by a gas current, or removed in undiluted form by pressure differences.

The earliest and simplest of all evaporators was the salt pan, heated by the sun, with natural convection removing the moisture. This type of evaporation is still practised in some parts of the world, sometimes on a very large scale. For instance, an evaporation pan covering 2000 acres, and built up from a concrete convergent spiral, has been erected in Mexico for the concentration of dilute natural brines. The amount of water evaporated is about 10 million tons per year, and an initial salt concentration of 1% increases to 30% as the solution flows from the inlet to the central outlet. This is equivalent to some 0·7 lb./sq. ft./day of water evaporated under ideal climatic conditions. Natural evaporation of this kind may also be used to cool process water. The supply of water at a suitable temperature for cooling or condensation is very often the final limiting factor to the output of a factory in any specific position. The problem of artificial cooling of water has been studied principally in connection with power station practice, but has a very definite importance in chemical works. In any normal plant, evaporation from a simple water surface—cooling pond—would demand far more space than is available, and some type of evaporative cooling tower will always be used. A modern concrete natural draught cooling tower (Fig. VI–1) may be 100–200 ft. high, with a base diameter about two-thirds of the height. Such a tower will cool a water feed from 90° F. down to 70° F. at a rate of 100–200 gal./sq. ft. of base area/hr. This is equivalent to an evaporation of

20–40 lb. of water per hour per sq. ft. of ground occupied. The working is by means of the chimney effect of moist warm air, and is clearly dependent upon the weather conditions prevailing from day to day. The figures given above are for normal climatic conditions in the south of England.

The limiting temperature to which water can cool in such towers will be given by the wet-bulb temperature Tw of the entering air-stream, and a measure of the tower efficiency will be given by the

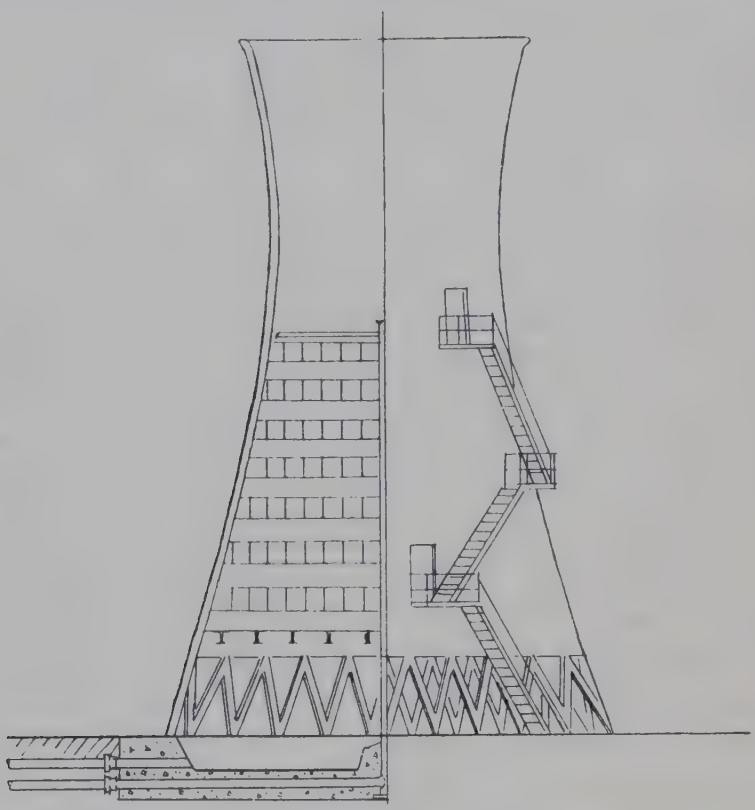


FIG. VI–1.—WATER-COOLING TOWER.

actual temperature drop $(T_1 - T_2)$ compared to the possible temperature drop $(T_1 - T_w)$. Towers of the type shown in Fig. VI–1 will give an efficiency on this basis of 40–50%, and have been criticised as relatively inefficient as compared to the packed towers used in gas-washing operations. By a careful arrangement of packing and the use of mechanical rather than natural draught it is possible to raise this efficiency to 75–90%.* The operation of these evaporating towers may be compared with gas-washing practice by noting that the mass transfer coefficients (pound mols/

* Hutchison and Spivey, *Trans. Inst. Chem. Eng.*, 1942, 20, 14.

hr. cu. ft./atm. press.) vary from 2·5 for natural-draught towers to as much as 15 for the best forced-draught towers. Air velocities vary from 2 ft. sec. up to 8 ft. sec., or 500 to 2000 lb./sq. ft. hr., with water rates of 2000 to 600 lb./sq. ft. base area/hr. Some criticism of the application of mass transfer coefficients to this problem has been made and a constant heat basis suggested.*

The water-cooling tower may be defined as a direct evaporator, and if the inlet gases are heated, then a large amount of water can be evaporated from a solution. If the solution is very corrosive and the temperatures required for evaporation are high, then a tower of this kind may be the only practicable plant for the process. The classic example of this process is in the Glover Tower of the sulphuric acid industry. This tower, besides recovering the dissolved nitrous gases from the "Chamber" acid, concentrates the acid from 60% strength up to 70% or more. The internal construction of the tower is a chequer-work of acid-resisting brick, down which the acid trickles; the lead sheath acts only to confine the liquid and gas. The hot gases, entering at about 700° C., are never cooled below about 150° C., and it is difficult to assess the value of vapour-pressure differences in promoting evaporation. The tower may be considered as a unit for heat transfer from hot gas to liquid, and it would appear that this rate is from 3–5 B.Th.U./cu. ft./hr./° F. in the packed section of the tower. A comparable figure for a water-evaporating tower of the type described above would be 100, but this is rather a misleading basis.

The long tradition of Glover Tower working shows the efficiency of the system, and has built up a regular market for acid of the strength normally to be obtained if all the "Chamber" acid is fed through the tower. It has recently been shown, however, that it is possible to concentrate sulphuric acid up to 96% in these towers, and thus obtain the second "normal strength" sulphuric acid of commerce. The rate of heat transfer diminishes somewhat to 2·5 B.Th.U./cu. ft./hr./° F. as might have been expected.† Evaporation of the remaining water in sulphuric acid has usually been carried out in large spray towers (Gaillard) or overheat pan evaporators (Kessler). These are now being replaced by drum concentrators (Fig. VI–2) in which the hot gases from producer-gas combustion

* Hirsch, *ibid.*, 1944, 22, 140.

† Cormack and Stubbs, *Trans. Chem. Eng. Group, Soc. Chem. Ind.*, 1948, 30, 49.

bubble through the acid. This inevitably causes spray formation, and an electrostatic precipitator must be used to strip the dilute acid spray from the waste gases. The very vigorous mixing makes for rapid heat transmission, and drum concentrators are reasonably compact. A double drum, of overall length 20 ft., 8 ft. diameter, will concentrate about 150 tons of 65% acid up to 92% H_2SO_4 in 24 hours. The vessels are of lead-lined steel, with a further lining of acid-resisting brick, and are protected from the action of the hot gases by the boiling acid. This is really an example of submerged-combustion heat transfer, as the gases are led direct from the

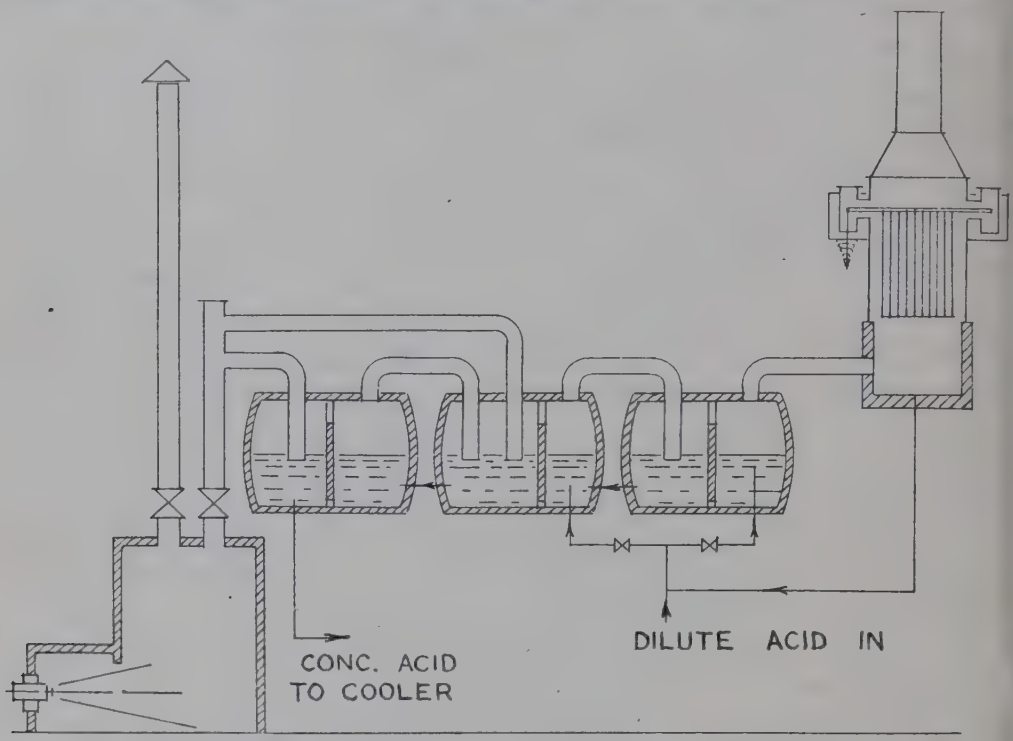


Fig. VI–2.—Drum Concentrator for Sulphuric Acid.

burner chamber into the liquid. It would appear that the velocity of the hot gas leaving the liquid surface is of the order of 2 ft./sec.

Sulphuric acid is an example of a crude commercial chemical of such corrosive nature that all plant must be of the simplest kind, and compactness and thermal economy must be sacrificed to the need for corrosion resistance. In the drum concentrator, however, the thermal efficiency is fairly high—4 lb. of water evaporated per lb. of oil fuel or solid fuel charged to producer. Another example of the use of submerged-combustion evaporators is to be found in the concentration of magnesium chloride. A simple burner in mild steel—frequently replaced—burns fuel oil under the surface

of the solution in a steel tank lined with acid-resisting brick, and gives an 85% utilisation of available heat.* A similar plant has been recommended by Swindin† for the concentration of spent "pickle liquor"—an acid solution of ferrous sulphate. It is clear that the use of fuel in this way involves the use of a high heat potential for a relatively low potential requirement. The same fuel could have been used to generate high-pressure steam at an even better thermal economy, the steam thus produced being fed to a pass-out steam engine, and the low-pressure steam obtained being used for the evaporation. This cycle of high-pressure steam → power producer → low-pressure heating steam is almost universally employed in chemical plants of any size. Wherever corrosion problems are not too difficult, the evaporation of solutions is carried out in steam-heated plant.

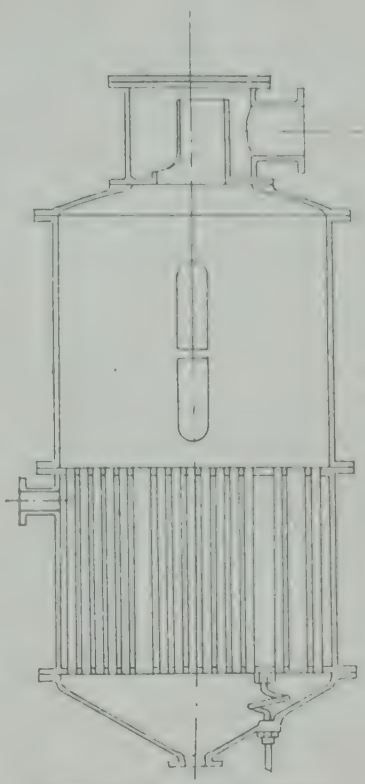


FIG. VI–3.—CALANDRIA EVAPORATOR.

Simple jacketed vessels, with steam condensing in the jacket while a solution boils in the pan, are hardly ever used, as the amount of surface associated with a given pan volume would be small, and the rate of heat transfer would be considerably diminished by the poor circulation of both steam and liquor. Some type of pipe surface is essential to obtain a large heating surface in a small volume, and it is necessary to ensure a rapid circulation of the boiling liquid. These requirements have been generally met by the "calandria" evaporator, as shown in Figs. VI–3 and VI–4. This evaporator was originally developed

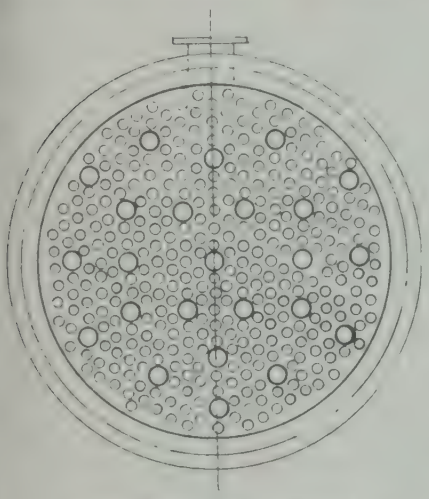


FIG. VI–4.—CALANDRIA EVAPORATOR.

* Wrege and Anstrand, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 12.

† *Trans. Inst. Chem. Eng.*, 1944, 22, 56.

in the sugar industry, but is now used in all branches of chemical technology. Steam is admitted to the calandria outside the tubes, the liquor boils on the inside, and a mixture of liquid and vapour is forced up the tube into the vapour space above by hydrostatic head. The vapour is disengaged in a space which is about equivalent to the volume of vapour formed per second, and the liquid returns by the various downtakes, of wider bore than the boiling tubes and scattered over the cross-section of the evaporator vessel (Fig. VI–5). The tubes are usually 3–4 ft. long, and the pressure at the bottom will be notably greater than at the top, due to the liquid head. This "submergence" effect is much more marked when the evaporator is working under vacuum, as the liquor is heated to a much higher temperature than pressure gauges in the vapour space would suggest, while the effective temperature difference causing heat flow is similarly diminished.

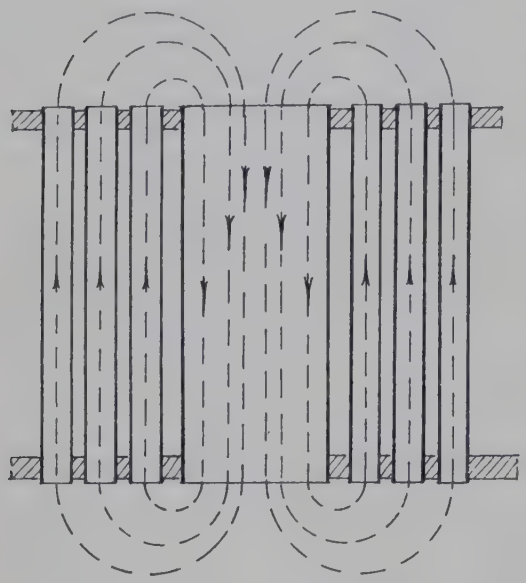


FIG. VI–5.—LIQUOR CIRCULATION IN EVAPORATOR.

The rate of heat transfer in such a unit is determined by "natural" circulation of the liquid, and to a lesser extent by the steam film and the tube wall. The main problem on the steam side is to sweep away the traces of non-condensable gas which are in practice always present, while the actual construction of the tube wall is of less importance than the avoidance of scale—a point that will be returned to later. The rate of circulation of the liquid does not appear to be notably effected by alteration from the small scattered downtakes of one manufacturer, to a large central downtake (Fig. VI–6a), or a side segmental tube (Fig. VI–6b). Other attempts to improve the circulation include a coned streamline form for the base of the evaporator (Fig. VI–6c) and a tilted calandria body (Fig. VI–6d).

It is a common property of many solutions that, in boiling, a more or less stable foam is formed. This must be given space to

break down, so that the vapour may be disengaged. An approximate figure for this disengaging space has already been suggested above, but actual surface conditions are also important. The vapour disengagement rate from a boiling liquid surface should not normally be more than 1 ft./sec. for non-troublesome materials at atmospheric pressure, and may drop to about one-tenth of this rate for (e.g.) crude sugar solutions. This will be equivalent to a maximum rate of 130 lb./sq. ft./hr. for water boiling at 100° C. Even allowing for

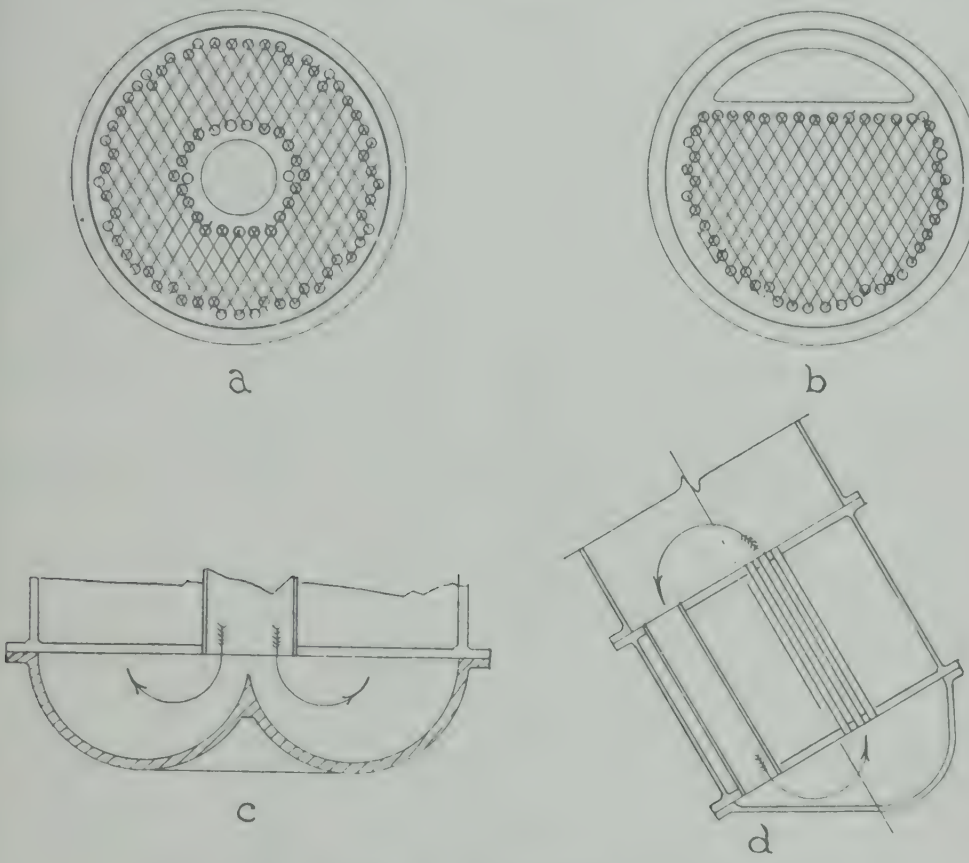


FIG. VI–6.—TYPES OF CIRCULATION IN EVAPORATORS.

disengagement space, it is common practice for spray traps, or "catchalls", to be installed at the top of an evaporator body. These traps are merely a series of baffles giving rapid changes of direction in the vapour stream.

An evaporator with a built-in calandria will be difficult to clean if scale deposits form. Formerly this was overcome by mounting the actual calandria as a suspended basket inside the evaporator body, and removing it as a unit for cleaning. The more modern tendency appears to be the construction of an "external" calandria,

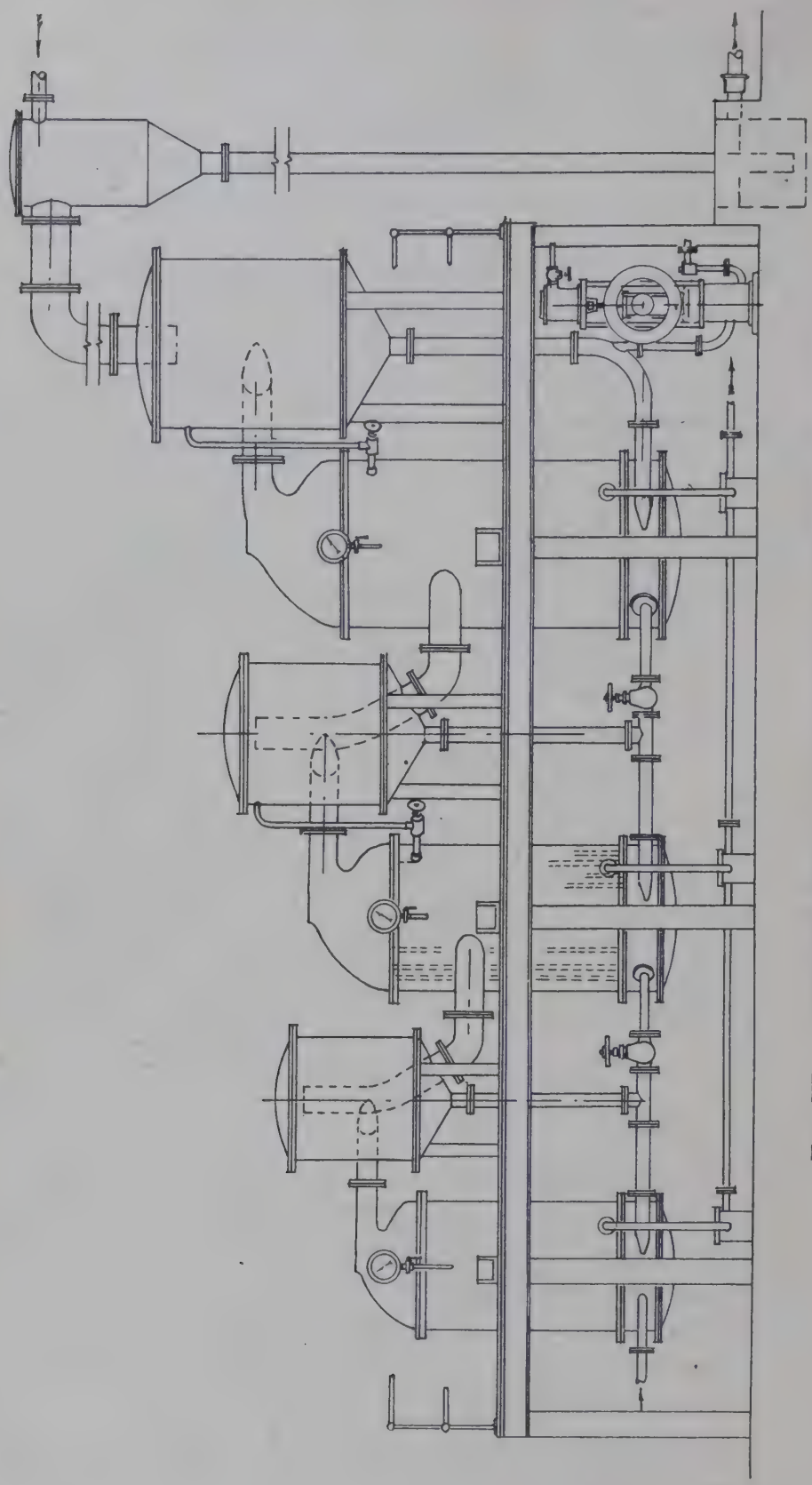


FIG. VI-7.—MULTIPLE-EFFECT EVAPORATOR WITH EXTERNAL CALANDRIA.

(Courtesy of Messrs. Blair, Govan.)

in which the boiling tubes are mounted outside the evaporator body, as shown in Fig. VI–7. The end-covers of the calandria are hinged and counterweighted for easy opening and cleaning. The position of these external calandria offers more resistance to fluid flow than that of the internal tubes, while at the same time the connecting tubes make the installation of some type of pump easy. A "forced-circulation" evaporator has therefore been evolved in which the liquid circulation depends almost wholly on the mechanical-pump

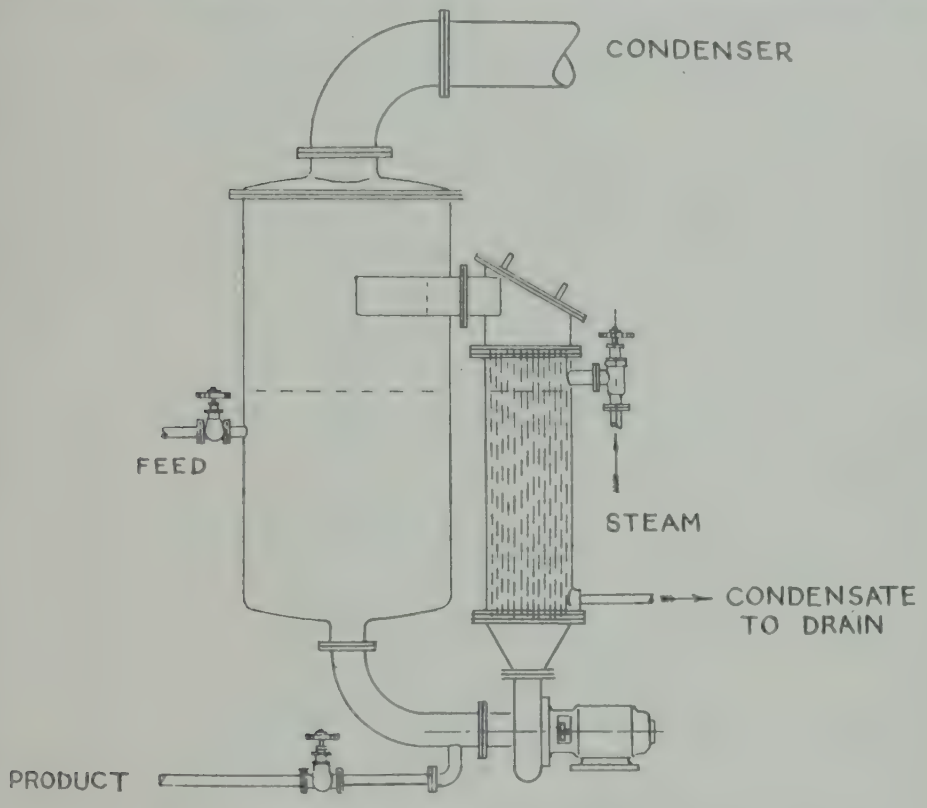


Fig. VI–8.—Forced-Circulation Evaporator.

system. A typical unit of this type is shown in Fig. VI 8, with external tubes, somewhat longer than those of the natural-circulation evaporator, and a large-disengaging "head". In these evaporators the rate of liquor circulation is such that little or no vapour formation occurs in the tube. The stream of liquid is heated under the pressure given by the circulating pump, and the discharge enters the body of the evaporator with a sharp pressure drop; considerable turbulence gives a rapid disengagement of vapour from the now boiling liquid. It has been suggested* that

* Boarts *et al.*, *Trans. Amer. Inst. Chem. Eng.*, 1937, 33, 363.

temperature measurements along an evaporator tube show a point of minimum temperature difference at which boiling commences, and that in forced circulation all the tube functions as a heater. It is doubtful if a very clear distinction can be made between " heating " and " boiling " tubes, while practice in this matter may vary from one industry to another. No very conclusive evidence has been put forward for more rapid evaporation—in pounds of water evaporated/sq. ft./hr. with these evaporators as compared to natural-circulation units. It is probable that the tubes may be kept more free from scale when forced circulation is practised.

There is no theoretical limit to the increase in rate of heat transmission through moving films of liquid as the fluid velocity increases, at any rate up to velocities approaching that of sound, but heat transfer to boiling liquids can only be increased up to a sharply defined maximum by an increase in temperature difference.* Nevertheless, the comparative ease with which good heat transfer can be obtained by boiling led to the development of the Kestner type of " climbing film " evaporator, in which boiling was utilised to obtain higher fluid rates over the heating surfaces than in the short tubes of the standard calandria. The tubes of the Kestner were 20–23 ft. in length, and the solution was admitted at the base; as it boiled, the vapour formed swept the residual liquid up the tube in " films ", at velocities of the order of 20 ft./sec. Fig. VI–9 shows the general arrangement of a Kestner evaporator, and brings out the important point that evaporation can often be completed by one pass through the long tube. This means that a thermally sensitive substance is only in contact with the heating surfaces for some 20 seconds. By contrast, in a typical standard calandria evaporator, the mean time of contact of a solution in evaporation may be up to an hour or more. Again, the concentrated solution emerging at the top of the tube of the Kestner may no longer form a stable foam, while the high velocity allows the vapour and liquid to separate in a cyclone separation head. The high degree of evaporation and the possibility of dry patches appearing on the walls of the heating tube appear to make the Kestner evaporator rather susceptible to scale formation, and the long tubes will then be difficult to clean.

The deposition of solids from the boiling solution is the most important problem in the actual working of an evaporator system.

* Cichelli and Bonilla, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 755.

Thus in sugar evaporation the rate of evaporation may drop by 50% over a period of 14 days' steady working, starting with a

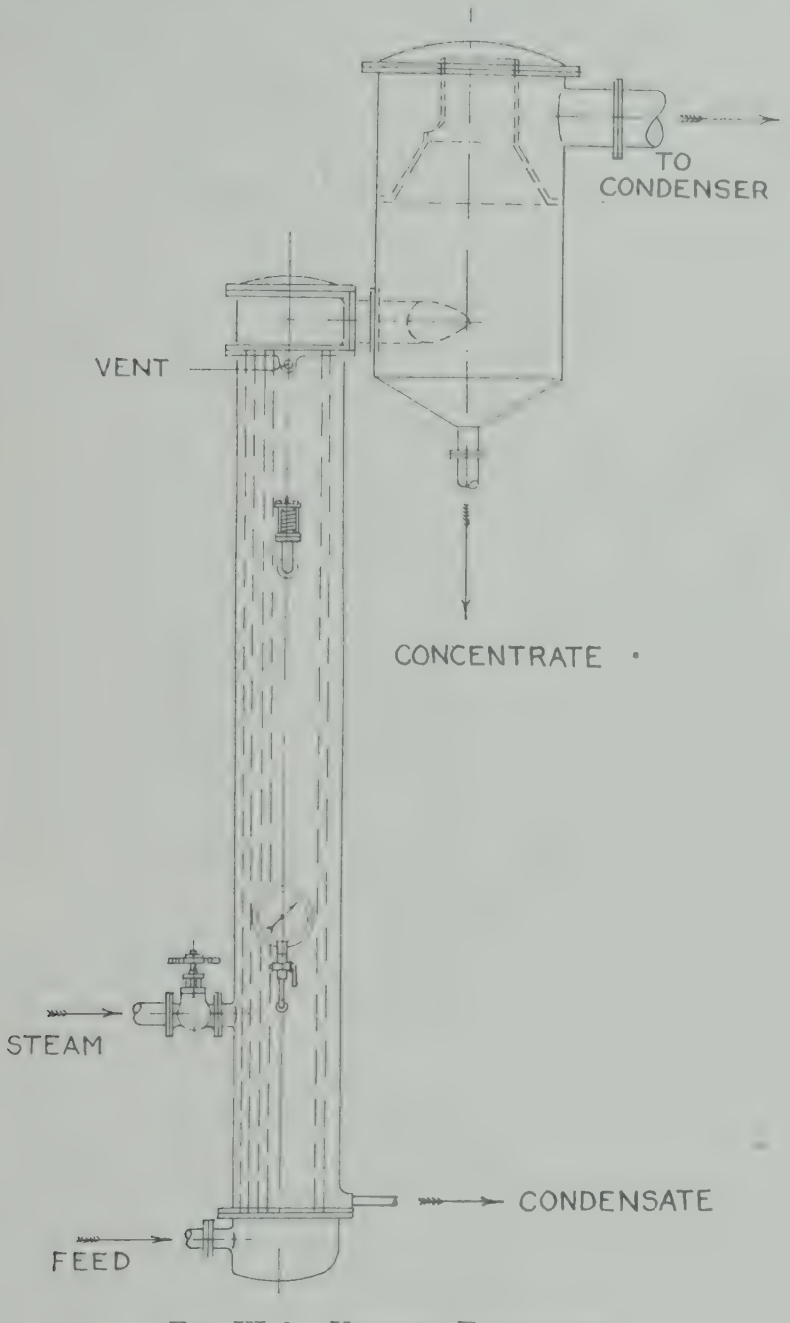


Fig. VI-9.—Kestner Evaporator.

cleaned vessel.* This is due to the deposition of scale on the inner side of the tubes, but there may also be some deposits on the steam side. Boiling with 10% caustic soda solutions, followed by

* Semple, *Trans. Inst. Chem. Eng.*, 1943, 21, 11.

hand-scraping, is required to remove the inner scale, while a dilute alkaline permanganate solution has been suggested for cleaning the outer steam side. Alternatively, some type of dilute acid wash, with an inhibitor to check attack upon metals, can be used for either side. A balance has to be struck between corrosive effects of cleaning solutions and complete removal of scale with enhanced heat transfer.

In the case of certain solutions it may be necessary to modify design in order to make scale removal more easy. Thus the Weir

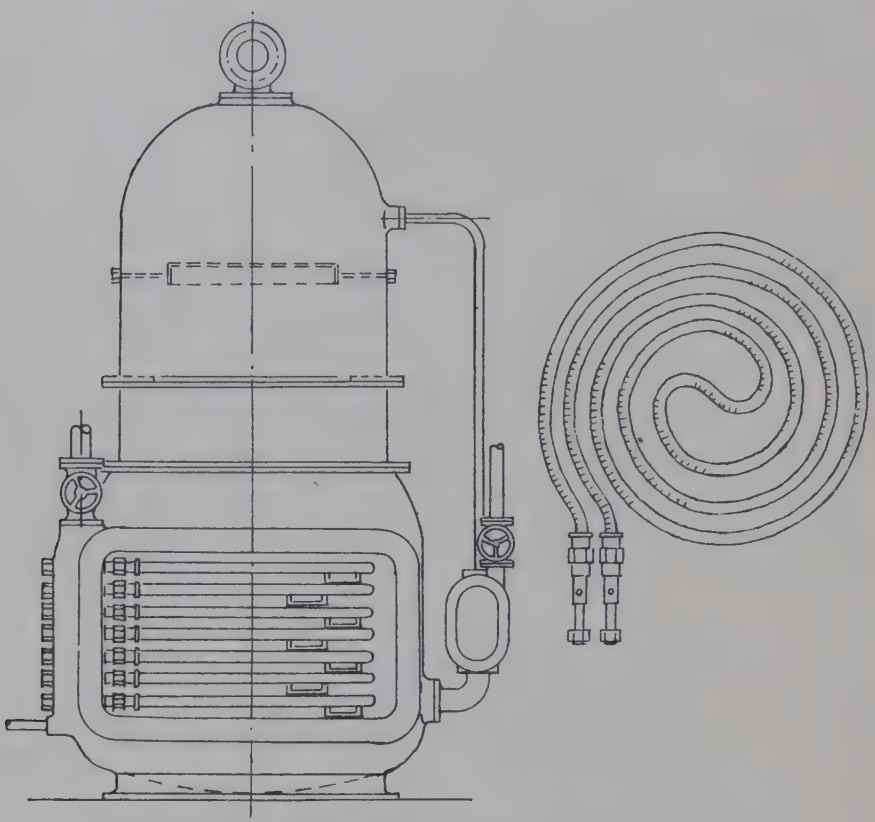


FIG. VI–10.—WEIR SEA-WATER EVAPORATOR.
(*Courtesy of G. and J. Weir, Glasgow*)

evaporator, built primarily for the evaporation of sea-water, has the heating surfaces in the form of pipe coils, with steam inside. These coils, as shown in Fig. VI–10, are in scroll form, and can be withdrawn and cleaned individually. Alternatively, by passing cold water through the hot coils, the metal contracts, and the hard shell of scale is cracked and drops off. Coils can also be used for very viscous liquids which will not flow properly through the tubes of a calandria. A typical example is found in the coil-heated vacuum pan for "finishing" sugar evaporation. The viscous

syrup holds a large proportion of sugar crystals in suspension, and heat-transfer rates are generally low. In an endeavour to improve this a design of evaporator shown in Fig. VI–11 used the coils for stirring as well as for heat transfer. This evaporator was actually

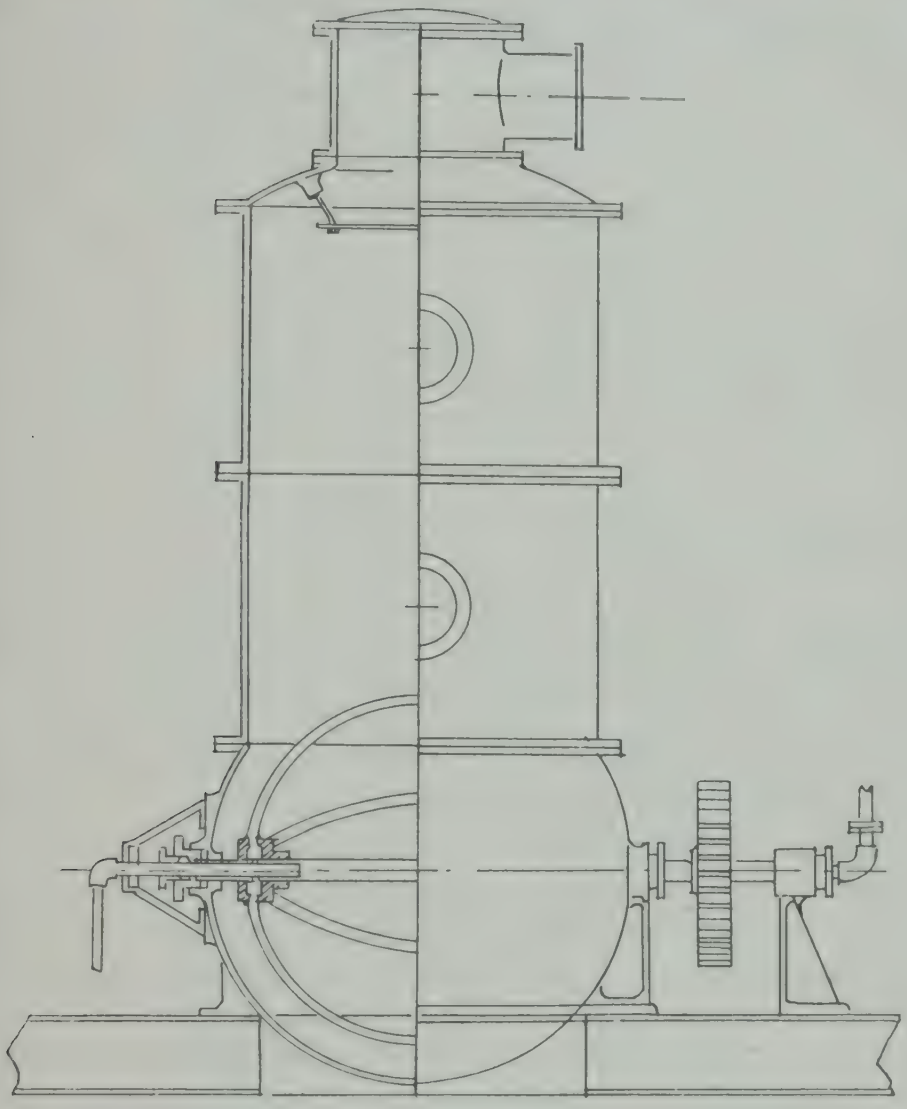


Fig. VI–11.—Evaporator for Concentrated Solutions.

built for tannin solution, and the great height of the evaporator was necessary because of the excessive foaming of the solution.

The output of a steam-heated evaporator is proportional to heat-transfer rates, which in turn are determined by temperature drop, arrangement of heating surface, and the specific properties of the solutions being evaporated. The effective temperature drop in an

evaporator is usually less than the figure suggested by the relative pressures in the steam and vapour spaces. This is due in the first instance to the submergence effect already referred to; in a typical case, suppose a 4-ft. tube filled entirely with water, and with the vapour space above at 2 in. mercury pressure (2·3 ft. water). Then the effective pressure at the base of the tube will be 6·3 ft. of water, and the boiling-point will be raised from 101° F. at the top to 137° F. at the bottom. If the steam in the steam space were at 5 lb./sq. in. abs. (162° F.), then the temperature difference would fall from 61 to 25° F. over the tube length. This drop is serious when the total temperature drop between steam entering and vapour leaving is small. In any typical evaporator problem, with steam entering the plant at a temperature of, e.g. 250° F., condenser water is available (in this country) at about 60° F., and will effectively condense vapour at any temperature down to perhaps 80° F. It is possible to make use of all this range (from 250° F.–80° F.) in one evaporator, but it will clearly be more economic to use the steam to boil a solution at about 212° F., to feed the vapour produced to a second evaporator where the solution boils at 170° F., and so on. This is the basis of "multiple-effect" evaporation, in which plant is multiplied but steam usage diminished.

An arrangement for multiple-effect evaporation is shown diagrammatically in Fig. VI–12. The vapours from each evaporator, or effect, are led to the steam chest of the next, while the solution flows in the same direction, losing water as it flows. This is the so-called forward feed, from the first evaporator, at the highest pressure, to the last. This dispenses with the need for pumps, but evaporates the most concentrated solution at the lowest pressure and temperature. If the liquid becomes very viscous in the last stages, heat transfer can be very poor, and in fact the limiting factor in evaporation by this method is generally the evaporation in the final stages. In some cases the liquor is pumped to a single-effect unit, at the highest possible steam temperature, to finish off the evaporation. This is done in both the sugar industry and in the paper-making industry. Backward feed, in which the liquor is pumped from one evaporator to the next highest in operating pressure, is sometimes practised; a typical example, also from the paper industry, used five stages for the evaporation of "black liquor", with two evaporators in parallel for the last, and in this case hottest, effect. As will be seen from the diagram, the condensate from each effect chest

can be passed down the line of evaporators, at each stage giving up a little more heat, so that the final discharge will only be at the lowest steam-chest pressure. This may not be always desirable, as the solution may give off non-condensable gas of a corrosive nature, and each case must be studied on its merits.

There is no theoretical limit to the number of evaporators which can be used in series between the original steam and the final condenser. A practical limit is soon set by the fact that good heat transfer can only be obtained with a temperature difference of more

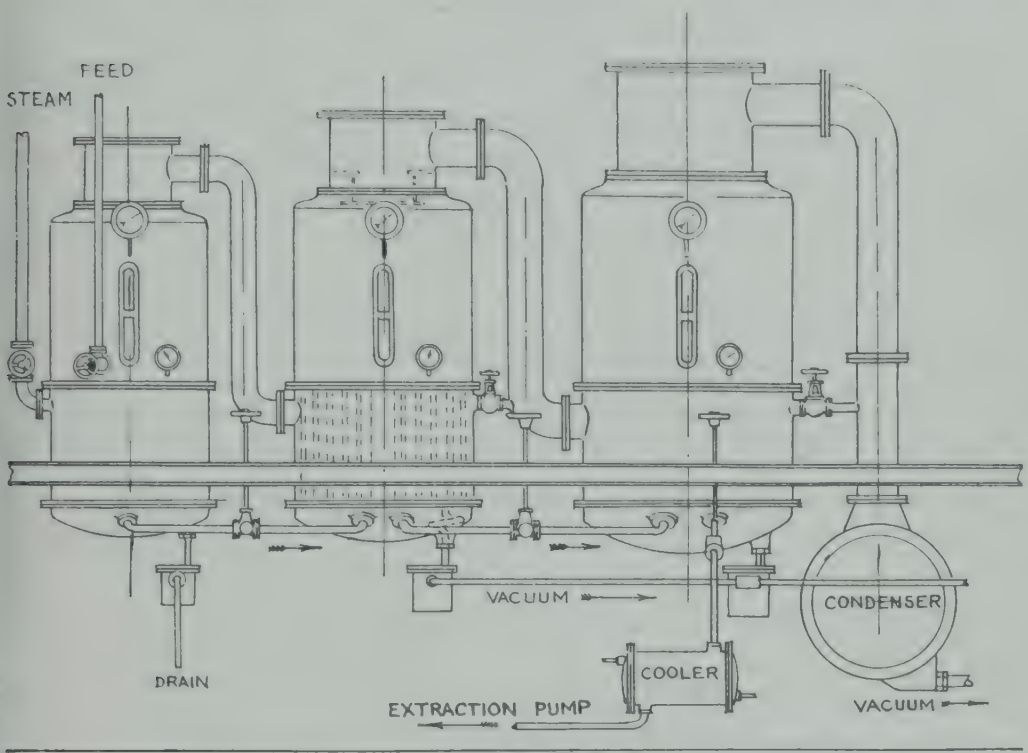


FIG. VI–12.—TRIPLE-EFFECT EVAPORATOR.

than 30° F., while an economic limit is reached when the plant cost exceeds the saving in steam. In a hypothetical and greatly simplified case, consider the plant required to evaporate 300,000 lb. of water per day, with steam costing 5 shillings/1000 lb., and each large evaporator unit costing some £1600/year, allowing for all capital charges. It may be assumed that each lb. of steam will evaporate $0{\cdot}8N$ lb. of water, where N is the number of effects chosen. If a working year is taken as 300 days, then the plant cost per day will be $\frac{£1600}{300}$, while the daily steam cost will be $£\frac{300{,}000 \times 0{\cdot}25}{1000 \times 0{\cdot}8N}$.

Then the total daily charge will be $£\left(\frac{16}{3} + \frac{75}{0{\cdot}8N}\right)$. The table below shows the variation of this cost:

N . . .	1	2	3	4	5
Daily charge (£)	99·3	57·7	47·3	44·8	45·7

As will be seen, the minimum cost is shown with four effects, but the change from three to four is small, and may be compensated for by saving in ground space or labour charges (usually small). It has been assumed in the example that the heat transfer rates will remain the same as the value of N is increased; if the temperature drop is less than 30° F. as a result of sub-dividing, this generalisation is no longer even approximately true. However, general practice in this country has led to the adoption of three- or four-effect plants. With the increased cost of fuel relative to plant which appears to be developing (1949) this practice may give way to five- or six-effect working.

As in most multiple-effect plants solutions are being concentrated, they will boil at temperatures above that of water at the same pressure. As the same vapour is being evolved, the steam from the solution will be superheated by the amount of this boiling-point elevation. In the steam space of the next effect the vapour will condense at its saturation temperature, or the effective heat drop between the two effects will be lowered. In the diagram of Fig. VI–12 it may be seen that the liquid in the second effect is boiling at 182° F., but the steam is at an effective saturation temperature of 180° F., so that the difference in temperature between steam and liquor in the third effect is diminished by 2° F. This is a relatively small drop, and the alteration may be much larger, according to the boiling-point elevation caused by the solute. It may be noted here that the principles of Duhring's Rule, described in the chapter on distillation, can be applied to determine the vapour pressure of solutions of which the boiling-point at two pressures is known.

The general principles of heat flow from a condensing vapour to a boiling liquid have already been dealt with, and it has been pointed out that the overall coefficient H_a cannot easily be broken down into the component film and wall coefficients. As a result, almost all figures dealing with evaporator practice are given in the form of an overall coefficient. The first and most important variable here is the temperature difference, and the effect of this on heat transfer is

shown in Figs. VI–13 and VI–14. The steep increase of H_a followed by a tendency to level out as values of 30–50° F. of temperature difference are reached is characteristic of both normal calandria and coil-heated evaporators. The reasons for this have already been

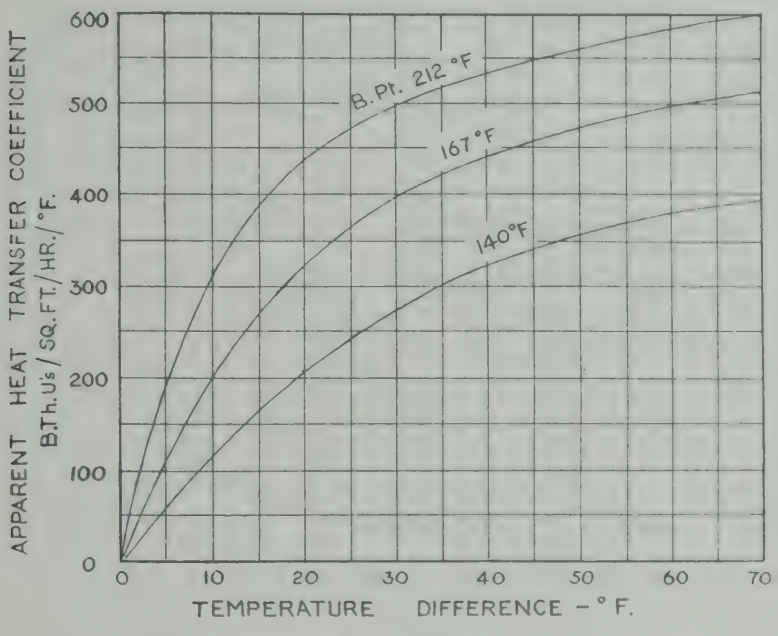


Fig. VI–13.—Heat-Transfer Rates: Condensing Steam—Boiling Water.

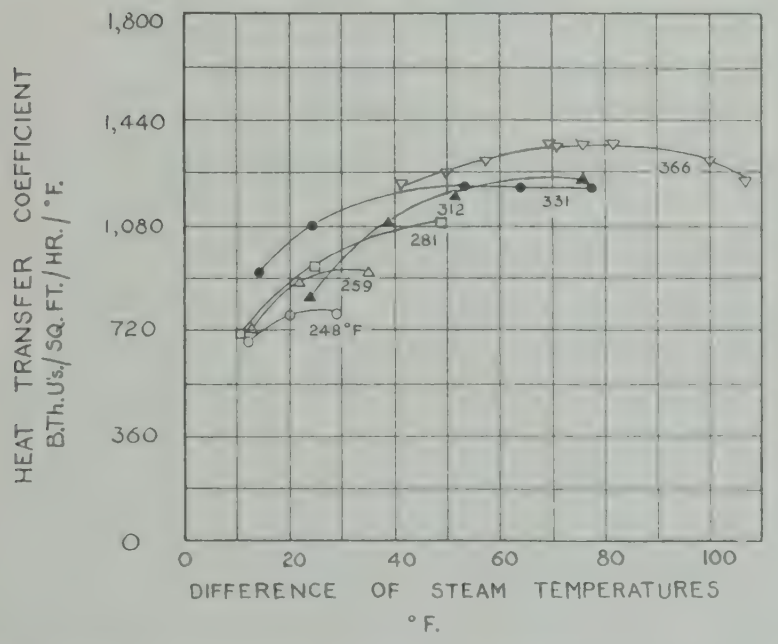


Fig. VI–14.—Heat-Transfer Rates: Condensing Steam—Boiling Sea-Water.

discussed in the chapter on heat transfer and are, of course, connected with the vapour " blinding " of the heating surface. There seems little possibility that any general formula connecting H_a with temperature difference will be established, especially when the changing conditions of the heating surface are taken into account. If the temperature drop is held steady, then the normal effects of solution viscosity and density tend to decrease the value of H_a, a decrease which is complicated by the fact that most evaporation plants work on forward feed and deal with the most viscous solutions at the lowest temperature. The data set out in Figs. VI–13 and VI–14 were for (*a*) distilled water and (*b*) a dilute solution—sea-water.* It has been shown that the value of H_a changes from 190 to below 10 as the density of a boiling syrup changes from 1·23 to 1·38. In these circumstances it is impossible to predict the rate of heat transfer that will be obtained in any evaporation project. If a solution is being evaporated which does not change markedly in properties from one effect to the next, then the following figures may be taken as a guide to performance:

Effect	1	2	3	4	
H_a . .	500	400	250		Triple Effect.
H_a . .	500	455	375	250	Quadruple Effect.

For a more detailed analysis recourse must be made to published data or to actual measurement.

The figures above are conservative, as higher results have been claimed for most special types of evaporator. Of late more attention has been paid to the steam side of the tubes, and it has been shown that proper arrangements to ensure rapid steam flow and removal of non-condensable gas can lead to considerable improvements in heat transfer.† Fig. VI–15 shows such an arrangement, with a wide steam inlet and a baffle round the downtake and gas vent. The drain pipe for condensate should be amply large enough to drain all liquid with a negligible weir head. The gases should be led off to the final condenser by a valve which is regulated to give a temperature difference between vent gases and inlet steam of 3–4° F. Greater differences will indicate inadequate venting, and lower, an unnecessary loss of steam. It is always possible to alter operating conditions slightly and attempt to obtain better working figures. For example, it has been shown that a standard calandria gives its

* After W. L. Badger, "Heat Transfer and Evaporation."

† Semple, *loc. cit.*

best performance when the liquid level is maintained at a static head about one-third of the way up the tubes. This may not be correct for all types of solution.

A preliminary calculation of the steam required, the weight of water evaporated in each effect, and the heating surface required can be based upon certain general assumptions. It is necessary to decide arbitrarily that some N effects will be required, and that for simplicity of construction these evaporators should all be the same size. This assumption of equal areas in each effect may not be the most desirable from the standpoint of minimum heating surface,*

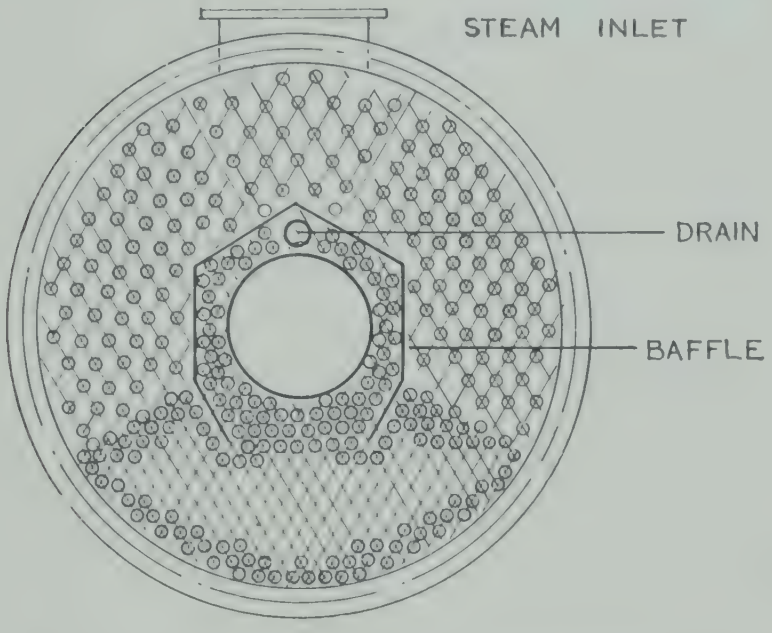


Fig. VI–15.—Calandria for Rapid Steam Circulation.

but the divergence is small and the ease of construction greatly increased. It follows from this assumption of equal areas that, as the amount of heat passing from one effect to the next is nearly constant, then the value of $H_a \times \Delta T$ will also be constant, where ΔT is the temperature drop from vapour to boiling solution. It may also be assumed that the loss of heat by radiation and convection will be negligible, and that the sensible heat of superheated steam can be disregarded as a set-off against this convective heat loss.

Example. *A triple-effect unit is to concentrate 22,500 lb./hr. of a solution containing* 10% *of solids up to a final concentration of* 30%

* Bonilla, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 529.

by weight. The feed is at 100° *C., and saturated steam is available at* 16 *lb./sq. in. ga.* (122° *C.). Condenser-water temperatures allow a final pressure of* 100 *mm. Hg, at which pressure the final solution boils at* 56° *C. Forward feed is to be used, and all condensates taken through to the final steam chest. Calculate the approximate heating area required, on the assumption that* H_a *will be* 500, 400, *and* 200 (*B.Th.U./sq. ft./hr./° F.*) *for first, second, and third effects respectively.*

(*a*) *Total heat drop* = 122° − 56° *C., or* 66° *C. Ratio of* ΔT *will be* $\frac{1}{500} : \frac{1}{400} : \frac{1}{200}$ *or* 2 : 2·5 : 5. *Division of* 66° *C. on this ratio gives* 14°, 17·5°, 34·5° *respectively, or the characteristic temperatures in each effect will be*

Steam . .	122°	108°	90·5° (*C.*)
Liquor . .	108°	90·5°	56·0° (*C.*)

But a solution containing 10% *of solids, boiling at* 108° *C., will give superheated steam, saturated at* 105° *C.* (*arbitrary value*). *Thus the real temperature drop in the second effect is only* 14·5°; *a similar diminution gives a real temperature drop of* 31·5° *in the third effect, and the real overall drop is only* 60° *C. Dividing this in the ratio* 2 : 2·5 : 5 *gives values of* ΔT *as* 12·5°, 16°, *and* 31·5°, *with a temperature distribution of*

Steam . .	122°	106·5°	87·5° (*C.*)
Liquor . .	109·5°	90·5°	56·0° (*C.*)

(*b*) *Let* W_x *be the weight of steam used, and* W_1, W_2, W_3 *the weights of vapour formed in each effect. The total weight evaporated per hour will be*

$$22{,}500 - \frac{2250 \times 100}{30} \text{ or } 15{,}000 \text{ lb.}$$

$$\therefore\ W_1 + W_2 + W_3 = 15{,}000 \quad . \ . \ . \quad (1)$$

The system may be represented by Fig. VI–16. Inserting appropriate latent heats, etc., and taking a heat balance over Effect 1.

$$945\ W_x = (22{,}500 \times 9{\cdot}5 \times 1{\cdot}8) + 960 W_1$$

$$\text{or } W_x = 408 + 1{\cdot}016 W_1 \quad . \ . \ . \ . \ . \ . \ . \quad (2)$$

Similarly, for Effect 2,

$$963 W_1 + \{19 \times 1{\cdot}8 \times (22{,}500 - W_1)\} + (W_x \times 15{\cdot}5 \times 1{\cdot}8 = W_a \times 980$$

$$\text{or } W_2 = 0{\cdot}978 W_1 + 808 \quad . \ . \ . \ . \ . \quad (3)$$

Effect 3:

$$983W_2 + \{34{\cdot}5 \times 1{\cdot}8(22{,}500 - W_1 - W_2)\} + \{(W_1 + W_x) \times 19 \times 1{\cdot}8\} = 1018\,W_3$$

$$\text{or } W_3 = 0{\cdot}892\,W_1 + 2120 \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Solving the equations 1–4 *gives*

$$W_1 = 4210,\ W_2 = 4920,\ W_3 = 5870,\ \text{and } W_x = 4673$$

(*c*) *The total heat flowing across the heating area* A_1 *in Effect* 1 *will be* $945 \times W_x$ *or* $4{\cdot}41 \times 10^6$ *B.Th.U./hr. As the value of* ΔT *is* 22·5° *F., and* H_a *is* 500 *B.Th.U., etc., the value of* A_1 *is* $\dfrac{4{\cdot}41 \times 10^6}{500 \times 22{\cdot}5}$ *or* 394 *sq. ft.*

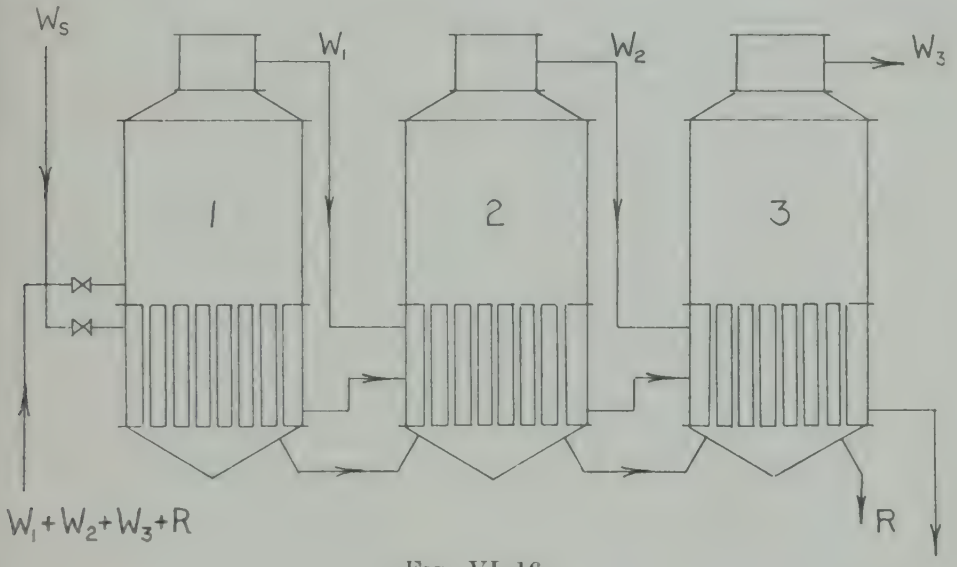


Fig. VI–16.

The total heat flowing across A_2 *in the second effect is* $963W_1 + 28W_x$ *– the flash heat of the solution is introduced directly. The heat flow is therefore* $4{\cdot}18 \times 10^6$ *B.Th.U./hr., with* $\Delta T = 29°$ *F. and* $H_a = 400$.

$$\therefore\ A_2 = \frac{4{\cdot}18 \times 10^6}{400 \times 29} = 362\ \textit{sq. ft.}$$

Similarly

$$A_3 = \frac{5{\cdot}14 \times 10^6}{200 \times 56{\cdot}5} = 456\ \textit{sq. ft.}$$

These three areas, 394, 362, *and* 456, *are rather far apart, and it will be necessary to adjust the assumed values of* ΔT. *Changes in the relative heat load for a small adjustment will be small and within the*

limits of accuracy of the method. If these heat loads are assumed as unchanged, then revised trial values of ΔT can be inserted in the equations to determine A_1, A_2, and A_3. Taking 12°, 14°, and 34° C. as the new values (21·5°, 25°, and 61·5° F.) the values of A_1, A_2, and A_3 become 407, 414, and 419 sq. ft. This is quite near enough for design purposes, and the evaporators will be built with 420 sq. ft. in each effect.

The method is lengthy and not very accurate. Methods have been proposed to cut down the work involved, or to find a more accurate solution without making test balances, but these do not seem to improve greatly upon the straightforward heat balance.* In any case, the fundamental inaccuracy of the method is rooted in the values assumed for H_a. Unless these values can be established accurately from practice, the area values obtained by any method must be adopted with considerable caution.

The purpose of multiple-effect working is to cut down steam usage at the expense of plant complication. If it were possible to step-up the temperature and pressure of the vapour evolved from the boiling solution, this could be fed back to the steam chest for further evaporation. Such a device—vapour recompression—is increasing in popularity, especially where a surplus of high-pressure steam is available. In the diagram of Fig. VI–17 a supply of steam at 100 lb./sq. in. absolute is fed through a steam-jet compressor, and entrains almost its own weight of vapour at atmospheric pressure, to give a mixed saturated steam at 20 lb./sq. in. absolute and 228° F. This mixture will evaporate 1·8 lb. of water for each lb. of high-pressure steam fed to the evaporator. It will be noted that the temperature drop is only 16° F., and heat transfer will be poor; this is characteristic of many thermo-compressor devices, as the entrainment ratio, or ratio of high-pressure steam to vapour compressed, falls rapidly if any attempt is made to design for a large pressure rise in the vapour. At the same time, it is notable that a thermal economy equivalent to double-effect working is obtained in a single evaporator. It is also possible to work a large single evaporator at temperatures well below the normal boiling-point of the solution, and to recompress the vapour with high-pressure steam. In this way a thermally sensitive solution can be evaporated with a considerable degree of thermal economy and no exposure to

* See, e.g., Storrow, *Industrial Chemist*, 1948, 174 and 311; Ray and Carnahan, *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 253.

high temperatures—a combination impossible in normal multiple-effect working.

The high-pressure steam may be replaced by some type of mechanical compressor, but again these compressors are not very efficient, high compression ratios taking the vapour into the superheated region, with consequent waste of energy. However, an evaporation of about 50 lb. of water per kWh. can be obtained, and the system has been adopted for quite small evaporators, generating

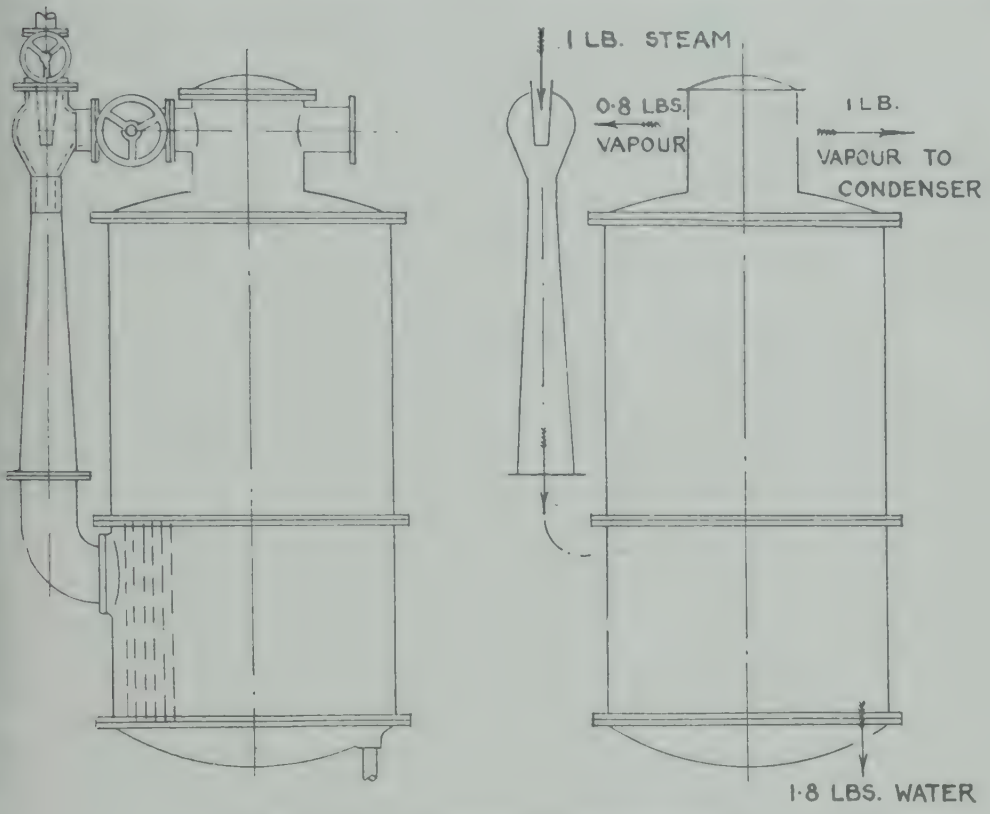


Fig. VI–17.—Vapour Recompression.

small quantities of distilled water from tap water or dilute brines. The principal use will be in the reduction of high-pressure steam down to a more normal operating pressure, but the method can be applied in a variety of ways. Thus, in the recovery of benzole from charcoal absorbers, a mixture of steam and benzole vapour can be used to generate steam at a lower pressure in a special evaporator unit. This low-pressure steam can be recompressed and fed back to the absorbers, with an economy in the use of steam amounting to 30% of the total used in benzole recovery.* In modern types of

* Hollings *et al.*, *Proc. Chem. Eng. Grp.* (*Soc. Chem. Ind.*), 1934, 16, 17.

thermal compression evaporators, a forced-circulation system is used to overcome the disadvantage of the low temperature drop.

Most evaporator plants are complicated in design, and considerable ingenuity has been used to construct such plant in corrosion-resisting material. The ideal constructional material from a general economic point of view is mild steel, perhaps with cast-iron evaporator bodies. The steel tubes for boiling are expanded into the tube plates, following general boiler-making techniques, and this introduces a mechanical stress at the top of the tube which may increase local corrosion. Nevertheless, it is rare to find special gland systems, although these have been introduced. Any good machineable metal may be substituted for steel and cast iron, and it has been found possible to manufacture calandria evaporators in such an intractable material as earthenware (for use with concentrated hydrochloric acid), while hard lead tubes or homogeneously lead-lined units have also been produced. When very corrosive liquids are being evaporated, however, it may be better to build the evaporator as a single tube. Thus a " falling film " evaporator for the vacuum concentration of sulphuric acid has been built from high-silicon iron in the form of a tube, 24 ft. long and 8 in. diameter, with a steam jacket.* It is possible that such steam-heated evaporators will ultimately replace the directly heated evaporators previously described.

The working of any multiple-effect evaporator, or single-effect operating under vacuum, involves the handling of large quantities of low-pressure vapour.

This vapour is almost always condensed in a jet condenser, in which a spray of condensing water comes into direct contact with the vapour. The condenser can be either of the counter-current type (Fig. VI–18a), which is economical in water, or the co-current type (Fig. VI–18b), in which the jets of water tend to increase the vacuum existing in the plant, acting as a supplementary vacuum pump. The amount of water required in these jet condensers will vary with design and water temperature, but will normally be 30–70 times the weight of water condensed.† The rate of heat transfer in the condensing space is of the order of 500 B.Th.U./cu. ft./hr./° F. It is essential to provide vapour pipes large enough to handle the low-pressure vapours, and linear velocities should not exceed

* *Chem. Eng. Prog.*, 1947, 43, 219.

† Tromp, *Int. Sugar Jour.*, 1946, 48, 119.

50 ft. sec. at atmospheric pressure, or 200 ft. sec. at a vacuum of 28 in. Hg.*

The final vacuum pumps which remove non-condensable gases from the system can be either of the " wet vacuum " type, taking both condensate, condenser water, and gas through a large valve system, or " dry vacuum ", removing only gas, and leaving the liquids to be pumped out by a special pump or run from the bottom

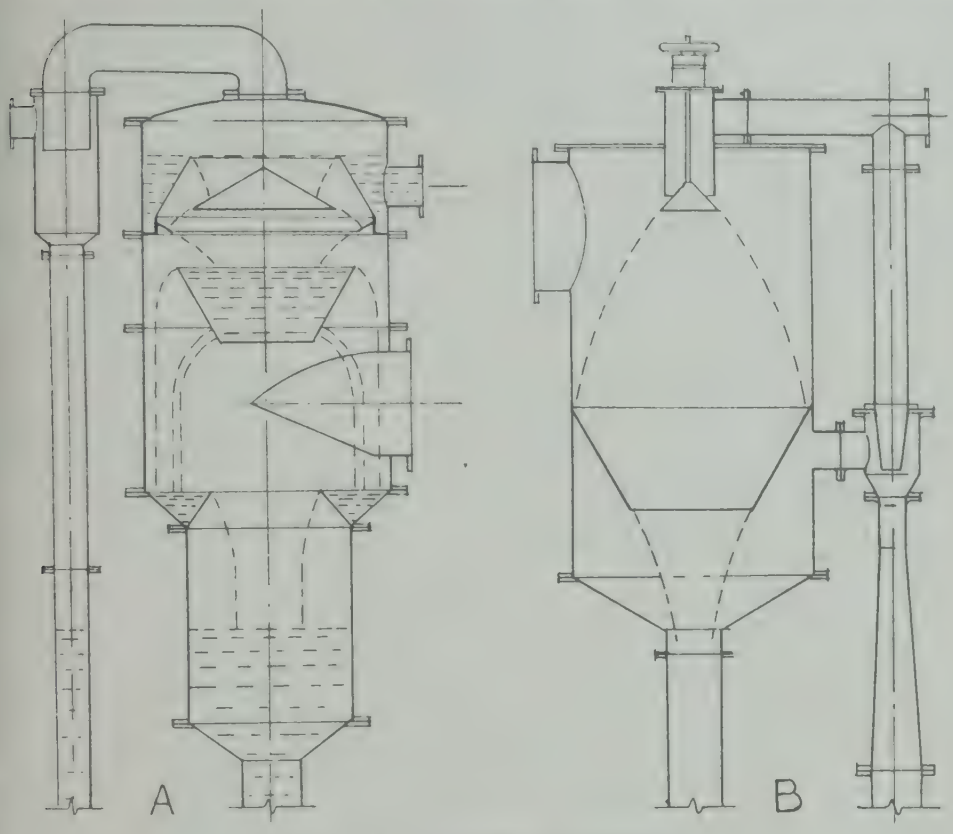


Fig. VI–18.—Jet Condensers.

of a barometric leg. These dry pumps must be built to handle up to 0·5% by volume of the total vapour passing to the condenser, together with air from the jet condenser water, if this is used. Alternatively, a capacity of 4 cu. ft. per lb. of vapour condensed in the last effect should be provided. As a rule, the pump is of the steam-jet type, and has already been described in the chapter on pumping.

* But consult, in this connection, Griffiths, *Trans. Inst. Chem. Eng.*, 1945, 23, 113.

CHAPTER VII

DRYING

The process of drying involves the removal of water (or other volatile solvents) from a residual solid by evaporation, as distinct from the mechanical process of draining by filtration or centrifuging. The utmost use should be made of these mechanical processes, because of the heat charges involved. In a simple instance a centrifugal will remove 2 tons of water from a ton of granular solid with a power cost of about 10 kWh.; the steam equivalent of this will be at most 300 lb. If the water is evaporated in a steam-heated dryer, the steam consumption must be at least 4480 lb. and may be much more, a margin which leaves room for very high capital costs in the case of the centrifugal unit. The case has been over-simplified, but shows that in general the drying plant will be designed for solids with a water content of the order of 50% or less.

The mass of wet solid will contain water in three forms:

(*a*) as free liquid in the " void spaces " of the mass;
(*b*) as liquid covering the surface of individual particles;
(*c*) as liquid in the interior of an individual fragment of solid, or combined with the solid in some other way.

In any drying process the rate of removal of water is determined by the difference between the vapour pressure of water at the surface of the solid and the partial vapour pressure of water in the atmosphere above. This difference, in turn, is determined by the comparative rates at which the water evaporates and at which heat is supplied to the solid–water mixture. The vapour pressure at the surface of the solid usually remains constant, under steady conditions, until all of the liquid in form (*a*) and some of that in form (*b*) has evaporated. Maintenance of a constant atmospheric humidity, as by steady air-flow or by mechanical pumping, will then lead to a constant drying rate. This is shown in the first portion of the curve in Fig. VII–1a, where rate of drying is plotted against moisture content. As the drying continues, the liquid film covering the solid surfaces grows

thinner and finally breaks in places, with a consequent drop in the rate of drying. The moisture content at which this drop in rate occurs is known as the critical moisture content. The form of the drying curve from this point will vary widely with the material being dried. It is common to find a further point of inflexion as the surface moisture disappears entirely, and this is followed by a steady fall in drying rate, with diffusion of water or water vapour to the surface of the solid as the controlling factor. The variation in this way is shown in the left of the curve of Fig. VII–1a. Where the amount of surface moisture is small, drying rates tend to follow the

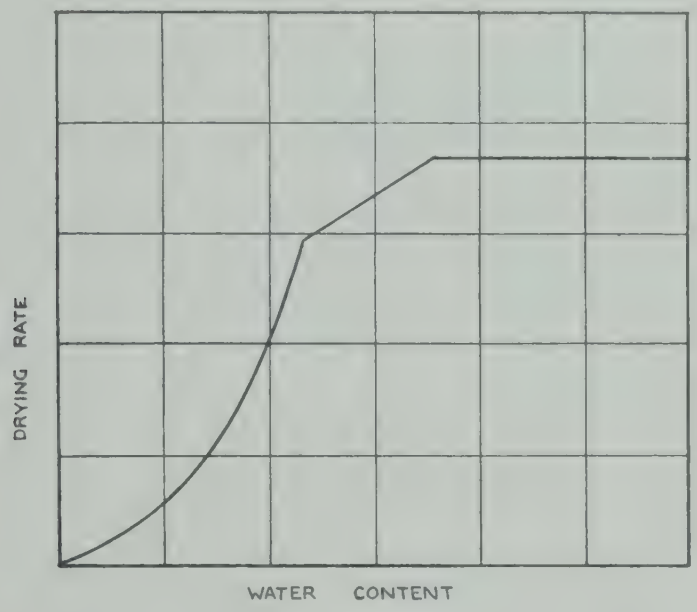


Fig. VII–1a.

line of Fig. VII–1b., in which the constant drying rate is followed by drying at a rate proportional to the residual water content. This allows of a mathematical treatment to determine the time of drying to any arbitrary moisture content.

Let the moisture content, in pounds per pound of dry solid, be W at a time T; then if the drying rate varies with the moisture content, $-\frac{dW}{dT} = KW$, where K is an experimental constant. Then it follows that $T = \frac{1}{K}\log_e \frac{W_1}{W_2}$, where W_1 is the initial moisture content and W_2 the content at time T. This has assumed that the solid can be dried to complete dryness under the conditions of test.

It may often be the case that the drying-rate line does not pass through the origin, but cuts the X axis at some point W_0, the equilibrium moisture content. Then the differential becomes $-\frac{dW}{dT} = K(W - W_0)$ and the integrated form will be

$$T = \frac{1}{K}\left(\frac{W_1 - W_0}{W_2 - W_0}\right).$$

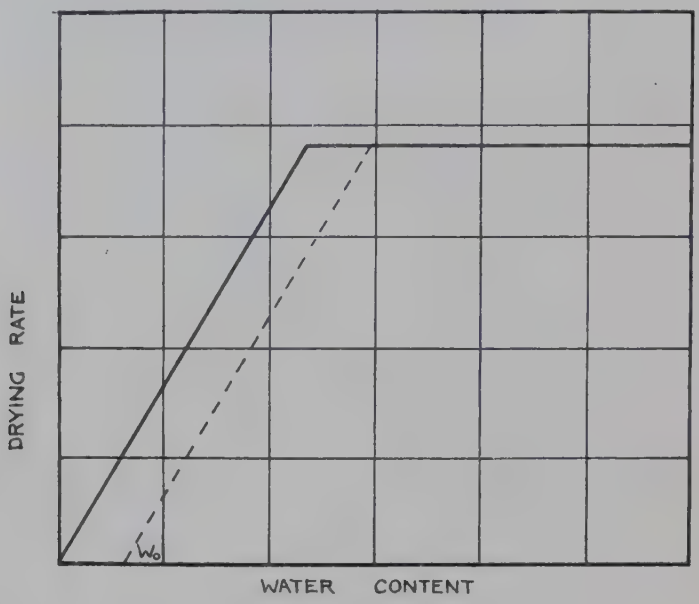


Fig. VII–1b.

Example 1. *Hanks of thread containing* 80 *lb. of water*/100 *lb. of dry fabric are being dried in a conveyor dryer. They dry at a constant rate of* 5 *lb. water/lb. of thread/hr. down to a* 50% *moisture content and then at a rate of proportional to water content. Calculate the time required to reach a water content of* 1% (0·01 *lb.* H_2O*/lb. of dry thread).*

The water evaporating at constant rate is 0·3 *lb. per pound of thread, and will take* $\frac{0{\cdot}3}{5}$ *hr. At this point the thread passes out of the constant-rate period, and*

$$5\left(\equiv -\frac{dW}{dT}\right) = K(0{\cdot}5), \text{ or } K = 10$$

In the falling-rate period

$$T = \frac{1}{10}\log_e \frac{0{\cdot}5}{0{\cdot}01} = 0{\cdot}39 \text{ hr.}$$

Total time $= (0{\cdot}39 + 0{\cdot}06)$ *hr., or* 27 *min.*

The drying rate is at all times determined by the speed at which heat can be transferred from the heating agent to the solid. This transfer can be by direct contact with heating surfaces, or by the passage of hot gases through or over the material. The simple hot-shelf dryer, in which heat passes up into trays of the material being dried, and moisture is removed by pumping or by convective air currents, is widely used for small quantities of solid. The vacuum-oven dryer, as shown in Fig. VII–2, is a cubical chest with a large door opening to give access to a series of steam-heated shelves.

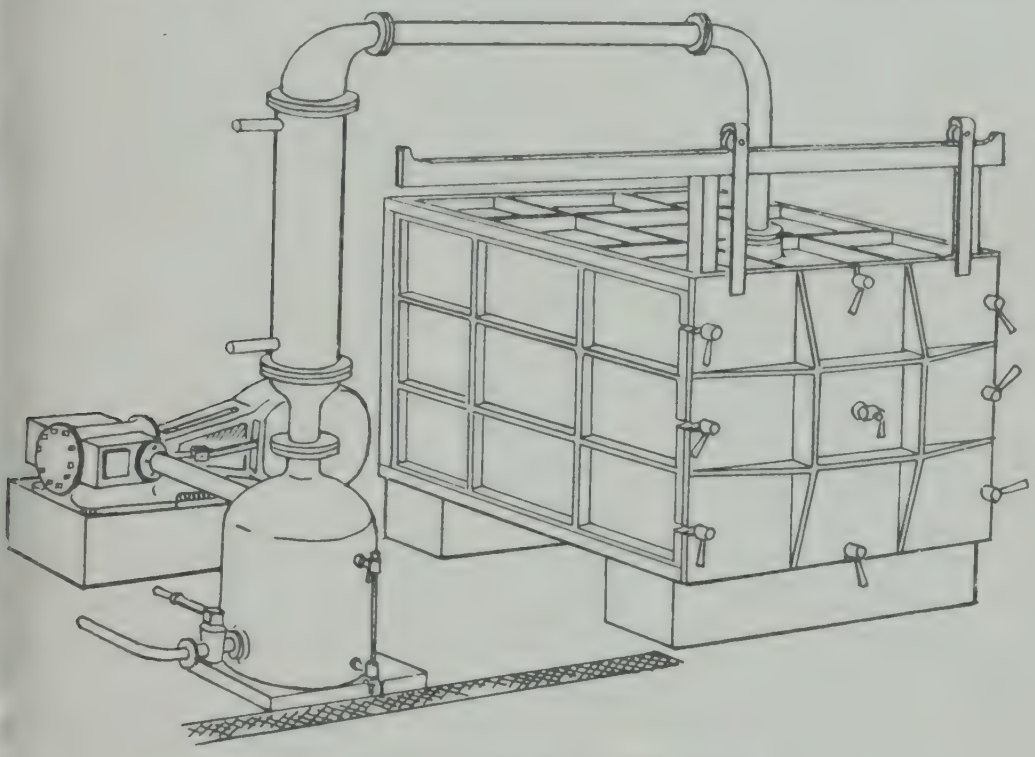


Fig. VII–2.—Vacuum Drying Oven.

These shelves are connected to a steam manifold at one end and a condensate discharge at the other. Material to be dried is loaded into trays which are arranged on the shelves by hand, and this need for direct manual labour restricts the size of vacuum ovens to about a 6-ft. cube. The trays can be of any suitable shape or size, but are customarily about 2 ft. wide by 6 ft. long and some 3–4 in. deep. They can be made in stainless steel, enamelled iron, or even glass, the last being peculiarly suitable for materials which are susceptible to chemical attack by metals. A number of small batches of different materials can be dried at one time in an oven, and this is particularly useful when small quantities of valuable

chemicals are being made. For good heat transfer it is necessary to see that the trays are flats and the shelves are not distorted, when the amount of water evaporated will be about 0·3 lb./sq. ft. of shelf surface per hour. This will vary, of course, with the material being dried, but is not usually outside the limits of 0·1 to 0·5 lb./sq. ft./hr. The thermal efficiency of the vacuum oven is high, about 80% of the steam heat being utilised in water evaporation. In the normal cycle of drying the oven is charged, the door closed, and the oven evacuated. Steam is turned on and the pressure maintained until the end of the drying period is near; the steam is then turned off and the drying completed by the stored-up heat of the oven. This avoids the heating of thermally sensitive substances to the actual steam temperature. It is customary also to slacken all locking bolts on the oven door during working and to rely on atmospheric pressure to hold the door in place. If a steam shelf bursts under pressure, the door opens, forming an automatic release for vapours which would be too much for the vacuum service.

The principal drawbacks of the vacuum oven are the small size, high labour charges, and the low rate of evaporation per sq. ft. of heated surface. If the material to be dried can be mechanically moved over the heating surface, heat transfer will be improved, and at the same time this movement can be arranged to convey the solid through the drying plant. A number of stirred dryers have been built for working on these lines. The simplest is the jacketed pan, as shown in Fig. VII–3. A flat-based cylindrical pan is fitted with a steam jacket at bottom and sides, and a scraper stirrer moves the pasty mixture over this heated surface to give evaporation rates varying from 1 to 3 lb./sq. ft./hr. When the drying is complete, a port at the base is opened and the same stirrer scrapes out the dry solid. A fair amount of power is required to drive the stirrer, an 8-ft. diameter pan taking 5 h.p. to stir a charge of 1 ton of meal, and the thermal economy at about 65% is lower than the vacuum-oven figure. Some materials cannot be dried in these stirred pans, as during the drying period they turn to a very stiff paste with poor heat-transfer characteristics. This "sticky stage" may stop the stirrer completely or roll the partly dried material into balls, which then dry very slowly. Similar difficulties may be encountered in direct rotary dryers, as described later. This illustrates the fact that the drying of any new material must be carefully studied, and that no general rules can be made.

The steam-jacketed dryer, with stirrer, can be built in various forms besides that shown in Fig. VII–3. It may be totally enclosed and put under vacuum, or the cylinder can be turned on its side, as in Fig. VII–4, and this appears to give an easier form of construction

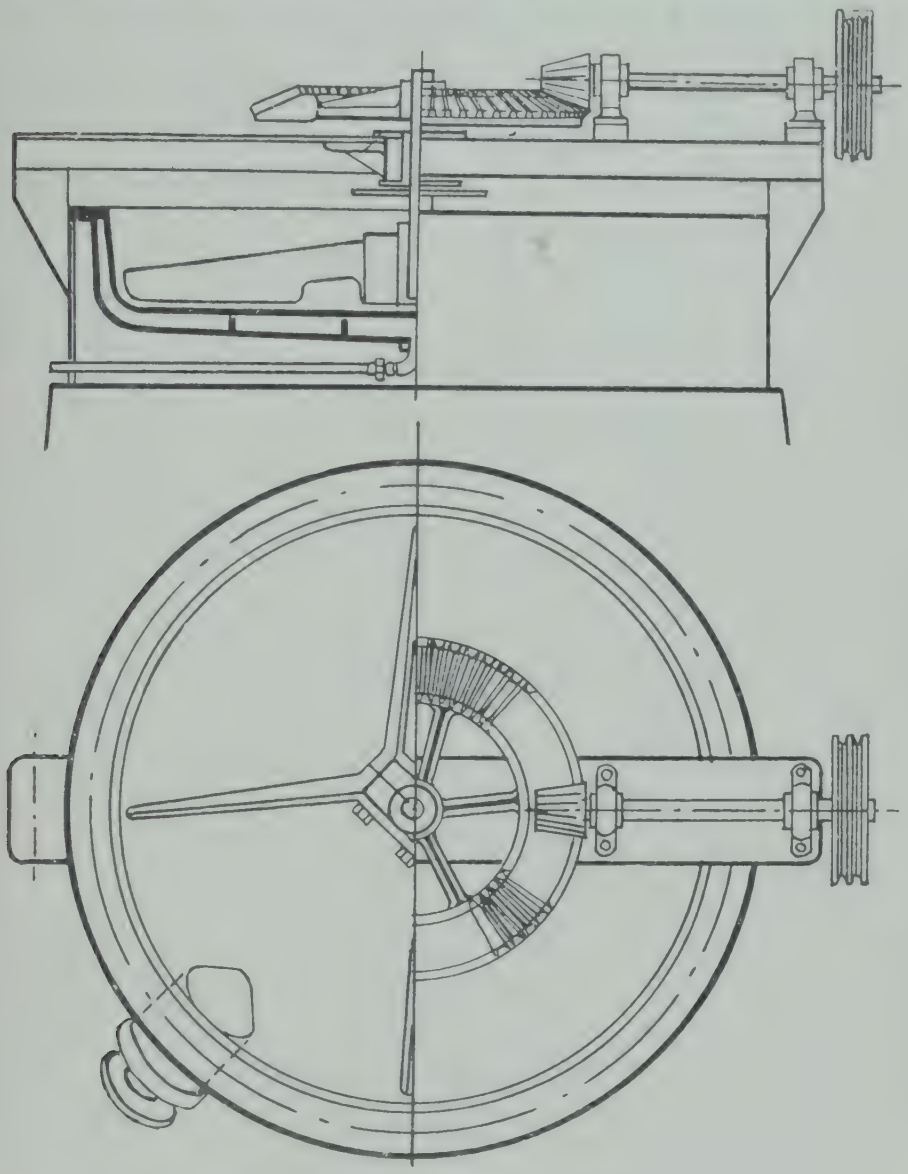


Fig. VII–3.—Open Pan Dryer.

for larger units. In the case of large amounts of stable mineral concentrates from flotation, all forms of enclosed vessel are abandoned, and the concentrate is spread over a hearth of heavy cast-iron plates heated from below by waste gas or steam. The material is raked along the hearth by "rabbling" arms, and rates of evaporation of the order of 3 lb./sq. ft./hr. are obtained in drying the

concentrates from 15% down to 6% of moisture. These hearths are said to be wasteful of heat and for most drying problems are inferior to direct rotary dryers.

Hearth dryers can be converted into direct dryers, with hot gases coming into direct contact with the solid, and these direct dryers are undoubtedly the most widely used drying plant. Heat must pass from the gas stream to the solid surface, at a rate which must be proportional to some function of the temperature difference. While free water remains on the solid, the surface temperature will approximate closely to the wet-bulb temperature of the air-stream. As the free moisture disappears the surface temperatures will tend to rise, and no regular relation between humidity and drying rate

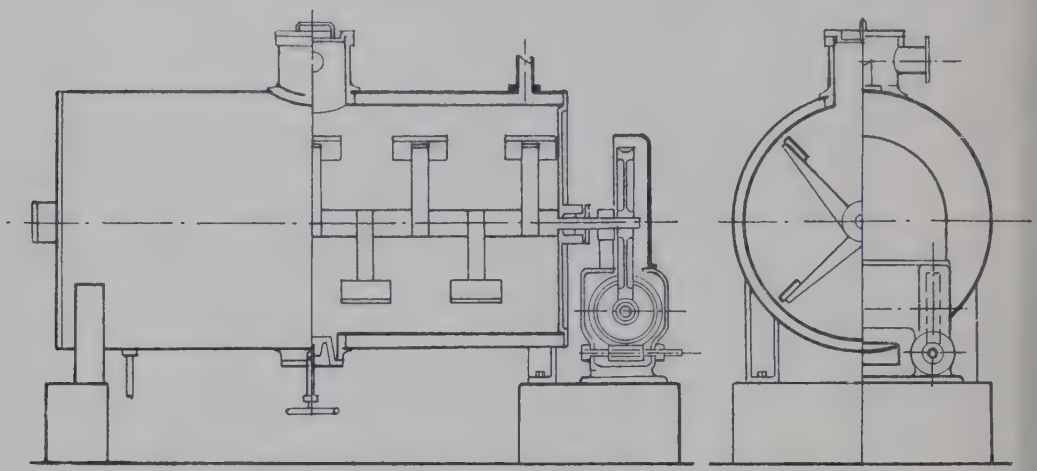


FIG. VII–4.—STIRRED VACUUM DRYER.

will persist. It is generally true, however, that while free water is present the rate of drying will be proportional to the difference between wet- and dry-bulb temperatures of the air-stream. For a wide range of substances being dried in a perforated-tray dryer, with the air-stream passing through the mass of material, it has been shown experimentally by Marshall and Hougen* that the rate of drying (S) in lb. water/lb. of dry solid/hr. followed the formula

$$S = G^{0.8} \cdot \Delta T \cdot \alpha$$

where G is the mass velocity of the air in lb./sq. ft./min.;

ΔT is the difference between wet- and dry-bulb temps. for the inlet air stream;

α is a specific constant for the material being dried.

* *Trans. Amer. Inst. Chem. Eng.*, 1942, 38, 91.

Unfortunately α varies from 3 with dense clays and sands up to 200 and above with fibrous material such as silk and cotton. If the surface of the wet material is considered as a free liquid surface, several investigators have proposed formulae for rates of evaporation. An early form, proposed by Hinchley and Himus* for water, gives the drying rate (R) (in lb. sq. ft. hr.) in terms of the formula

$$R = (0{\cdot}0063 + 0{\cdot}00084V)(p_A - p_S)$$

where V is the air velocity in ft./sec.

$(p_A - p_S)$ is the difference between aqueous vapour pressure at the liquid surface and that in the air-stream (in mm. Hg).

For air at 200° F., containing 0·005 lb. water per lb. of dry air, the wet-bulb temperature will be about 86° F. Then water exposed to the air will tend to reach this temperature, when p_A will be 32 mm. and p_S 6 mm. For a velocity of 10 ft./sec. over the surface

$$R = (0{\cdot}0147) \,.\, (26), \text{ or } 0{\cdot}38 \text{ lb./sq. ft./hr.}$$

The results given by this formula have been criticized by Powell and Griffiths† as being obtained with relatively small liquid surfaces and high edge effects. They are therefore on the high side, but small areas and eddy currents are the usual conditions obtaining in the tray dryers and the formula may well represent the maximum rate of drying in many cases. A comprehensive formula by Wade‡ may be consulted if non-aqueous liquids are being evaporated.

It is doubtful if any general formula can be applied to all cases of drying, even in the constant-rate period, and where the free moisture is gone and rates of diffusion from the interior of the solid determine the drying conditions, no rules can be applied. It is always necessary to obtain actual experimental data on the material itself, or on some closely allied substance.

Ordinarily directly heated shelf dryers, with various forms of air circulation, are cheap to build, and can often be constructed *in situ* from asbestos or steel-sheet casings around angle-iron frames. The shelves can be mounted on wheeled trucks and pushed through

* *Trans. Inst. Chem. Eng.*, 1924, 2, 57.
† *Ibid.*, 1935, 13, 175.
‡ *Ibid.*, 1942, 20, 1.

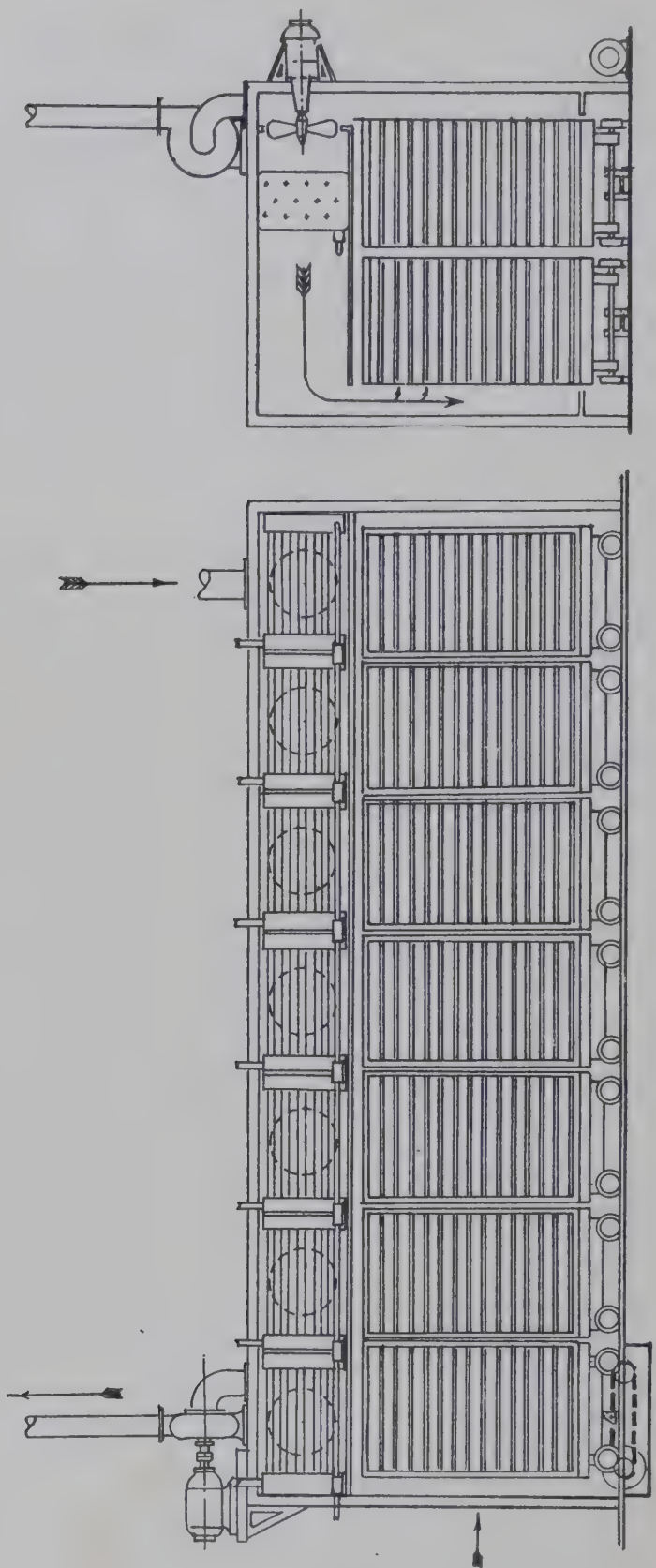


FIG. VII-5.—DIRECT TUNNEL DRYER.

a long tunnel dryer, as shown in Fig. VII–5. The circulating air is blown across the trays and over reheating tubes. Meanwhile a separate fan keeps up a steady flow of air counter-current to the trucks of drying trays. This recirculation of air greatly improves the thermal efficiency of direct dryers, as can be seen from a study of Fig. VII 6. Suppose the initial inlet air to contain 0·005 lb. (2·4 grains) of water per lb. of dry air. At a temperature of 60° F. it will be represented by the point A, and during the first heating by steam pipes conditions will change to some point B. Passage over the wet stock will cool the air to condition C—100° F. and 0·028 lb.

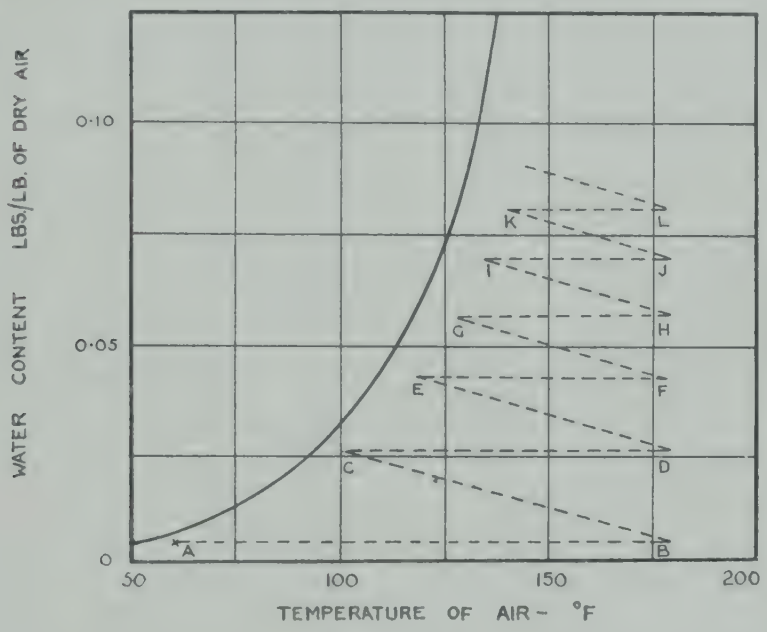


Fig. VII–6.

water per lb. of dry air. Single counter-current flow would remove the air at this point, with 0·023 lb. of water evaporated by each lb. of dry air. Continued reheating and recirculation will give the series of air conditions shown in D–F–G–H–I, while removal at the condition represented by the point I will give an outlet air at 140° F. containing 0·082 lb. of water per lb. of air. The effective heat usage in the dryer is represented by the latent heat of the water evaporated, and the ratio of this to the total heat leaving represents the actual dryer efficiency; in the case above single contact gives an efficiency of 70%, as compared to 80% with the recirculation system. The air will never reach saturation as represented by the firm line of the figure.

Tunnel dryers, with the trays on trucks, can have very high capacities, but stationary-shelf dryers are usually smaller, with manual discharge of trays, and suffer from the same disadvantages in this respect as the vacuum-oven dryer. Thermally sensitive or dangerous chemicals may be safely dried in this way; wooden trays on wooden supports, with all temperatures below 80° C., can be used to dry certain types of explosive. In most tray dryers the air passes over the surface of the tray, and this does not make for such rapid drying as in the case where the trays are perforated, with the drying air blowing through the bed of solid. The "through" dryers described by Marshall and Hougen (*loc. cit.*) were designed on these lines, but the trays were replaced by a wire-gauze belt, which conveyed the material through the dryer. These solids should ideally

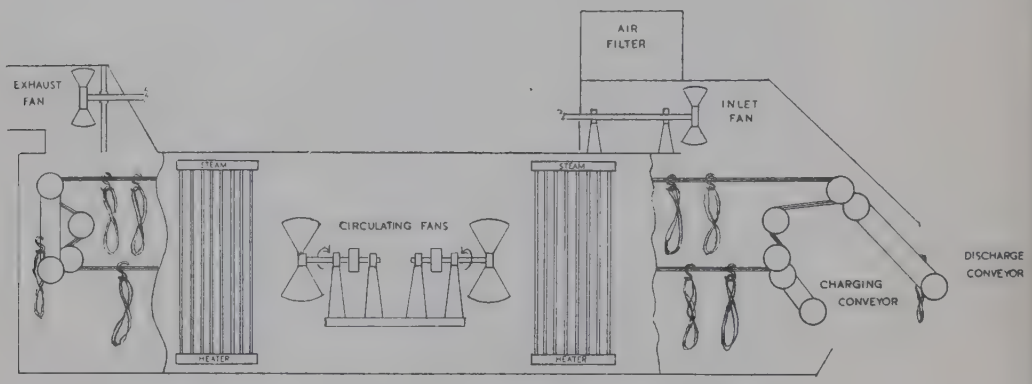


Fig. VII–7.—Continous Cotton Dryer.

be evenly divided, and in the above case the pasty solid was granulated in some way to give an even resistance to air-flow. The conveyor-belt system has been widely used in other direct dryers, and Fig. VII–7 shows a diagram of a cotton dryer, in which the hanks of wet cotton are fed in at the bottom front, conveyed to the back, lifted, and brought forward to the front for discharge. The material flow in such a unit seems at first sight to bring wet and dry cotton hanks into close proximity, but the air-flow is from top to bottom, with the hot air coming into contact with the top dryer layers before the wetter hanks underneath are reached.

The vigorous circulation and recirculation of air in such dryers calls for a considerable use of power. German drying practice* utilised curved-vane baffles to avoid eddy losses in the reversal of

* *B.I.O.S. Report* 1587.

air-flow. It was also suggested that a pulsating pressure—over about 8 in. w.g.—would break up stagnant air pockets and increase diffusion rates in a mass of powdered solid.

If the material does not pass through a sticky stage in processing, is free from very fine dust, and is to be dried in large quantities, a direct rotary dryer is the most efficient form of plant. Rotary dryers are long cylinders mounted on a slight slope, and revolve slowly as the material being dried and the hot gases pass through.

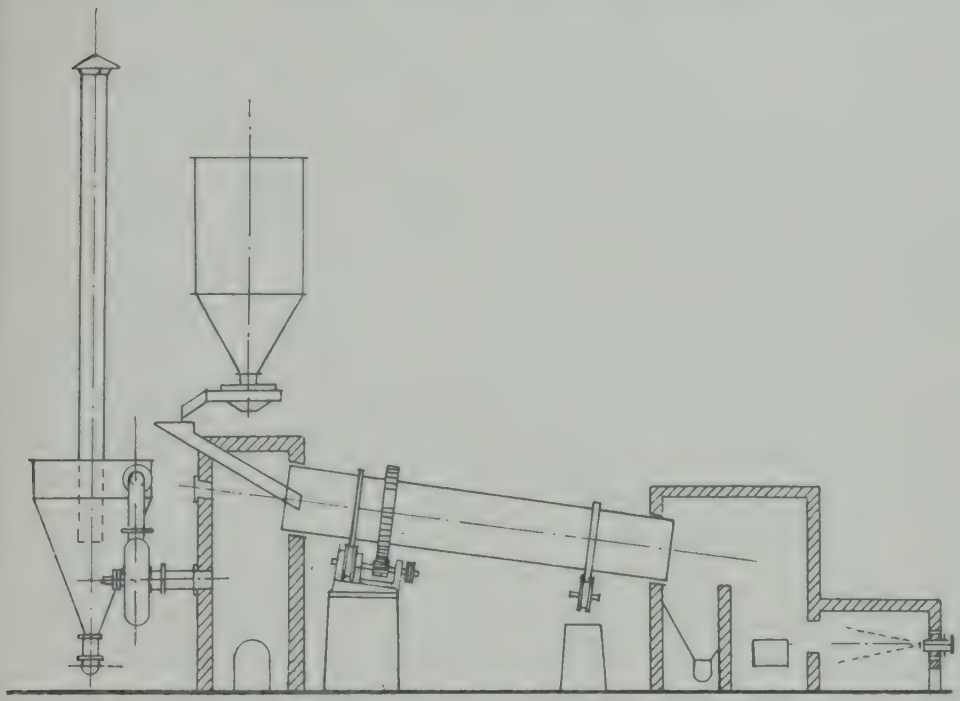


Fig. VII–8.—Direct Rotary Dryer.

Lifting devices on the interior of the cylinder raise the solid, which must be free-flowing throughout the process, and cascade it through the hot gas stream, which is usually flowing in counter-current. A typical rotary dryer is shown in Fig. VII–8; the solid advances down the dryer in an irregular spiral path, at a rate determined by the dryer slope, the speed of rotation, and the design of the lifting shelves. An approximate formula for the time of retention (T) has been proposed by Prutton* *et al.*

$$T = \frac{k \times L}{R \times D \times S}$$

* *Trans. Amer. Inst. Chem. Eng.*, 1942, 38, 123.

Here L is the length, and D the diameter, both in feet; S is the slope in inches per foot, and R the speed in r.p.m.; k is a value determined by the shelf arrangement, and increases from 3·3 to 4·5 as the number of simple lifting shelves is increased from 6 to 12. A small additional correction for air velocity was suggested, but is hardly justified in view of the approximate nature of the formula.

Then for a dryer of 7 ft. diam. by 56 ft. long, having a total slope of 21 in. from end to end, and a speed of rotation of 3·8 r.p.m., and with 8 shelves ($k = 4$), the retention time will be

$$\frac{4 \times 56}{3{\cdot}8 \times 7 \times 0{\cdot}375} = 22{\cdot}5 \text{ min.}$$

This is, of course, a mean retention time, as with all continuously working units—a theme that has been widely investigated in recent years.

The total amount of solid in the dryer should not, as a rule, occupy more than 25% of the total volume and figures of about 12% are usual. The surface exposed for drying is a matter for conjecture; each shelf is showering material as a curtain down from top to bottom of the cylinder, but these curtains are permeable to the air-stream and will expose more surface than a compact solid wall. It would appear desirable to base dryer design on volume of drying space, which is quite definite. As pointed out above, the solid will heat up to the wet-bulb temperature of the air-stream, and beyond this in the later stages of drying. If the temperature of the drying solid can be measured directly, the process can be treated as one of heat transfer between the air and the solid. Analyses of typical working figures suggest that heat-transfer rates of from 5–10 B.Th.U./cu. ft./hr./° F. can be obtained in these rotary dryers. There will be some variation with gas and solid velocities, but these are limited by the fact that gas velocities cannot exceed about 10 ft./sec. or solid entrainment and dust troubles become very marked, even with large solid particles of 0·25 in. size. While the surface exposed by finer solids is much greater than with these large fragments, the limiting air velocity is lower—1 to 3 ft./sec. with crystals of 0·05 in. size.

It is possible to base an estimate of dryer capacity on these figures. Suppose that the solid is fed in at 20° C. and leaves at 50° C., while the drying air flows counter-current, entering at 150° C. and leaving at 50° C. Then the logarithmic mean temperature

difference will be 51° C., and on a basis of 8 B.Th.U./cu. ft./hr./° F. the heat-transfer capacity will be 735 B.Th.U./cu. ft./hr. The dryer in the example above, 7 ft. diam. by 56 ft. long, has a volume of 2150 cu. ft., so that a total heat load of 1,580,000 B.Th.U./hr. can be expected. If the dryer is removing 5% of water from solid of specific heat 0·4, the heat load per ton of dry solid is some 160,000 B.Th.U., so that the capacity of the dryer will be 10 tons per hour. The volume of solid involved in a working period of 22·5 min. will be 120 cu. ft. (on a basis of 70 lb./cu. ft. for loosely packed solid), while the air volume works out at some 130 cu. ft./sec. at N.T.P.; these figures are well inside the limits suggested for drying conditions. The dryer will, in fact, be lightly loaded, as would be expected from the relatively low air temperatures.

This method gives only an indication of capacity, as many other factors may play a part. As with filtration, all substances have specialised characteristics, and experimental details must be obtained with the material itself before any exact design can be attempted. It is particularly necessary to ensure that the solid does not stick in the dryer, as any tendency to accumulate on the shelves makes the rotary dryer unworkable. Another factor which has to be considered is the design of the lifts and baffles in the cylinder. Some typical designs are shown in Fig. VII–9; the steam jacket (a) or steam drum (b) add direct heating to the hot gas effect, while the cross shelves of (c) are claimed to give better distribution than simple cascading from the normal peripheral shelf. The design of (d) is notable as an attempt to avoid cascading altogether, as the drying air passes up from the louvres forming the inner shell of the dryer and through the bed of drying solid. These louvres form a cone section with the narrower end at the feed, while the outer drum is on a horizontal axis; a valve cuts off air from the louvres which are not covered by the solid layer. The quartered section (e) is typical of a number of designs in which the cylinder is divided into sections.

Where very high temperatures of the inlet air—up to 1000° C.—are permissible, a double-shell dryer has been widely used. This is designed as shown in Fig. VII–10, and it will be seen that the hottest gas is not in direct contact with the material to be dried, which is all in the outer shell. At the same time, problems of heat loss from the hot walls and of mechanical design for hot metals are considerably reduced, as the outer shell walls never reach higher temperatures than about 200° C.

The power requirements for rotary dryers are not high; a dryer 40 ft. long and 5 ft. in diameter takes 10 h.p. to turn it at 2 r.p.m. while the fan for air circulation requires another 4 h.p. A dryer

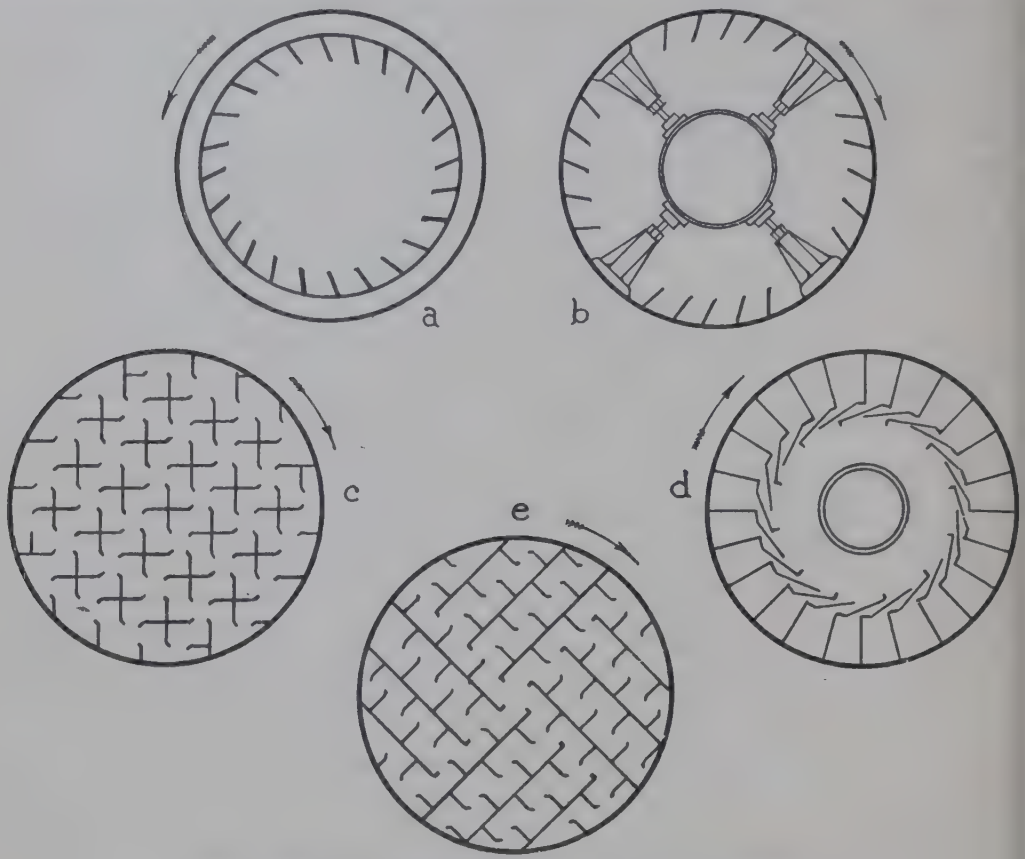


Fig. VII–9.—Shelf Design in Rotary Dryers.

45 ft. by 9 ft. requires 24 h.p., with 10 h.p. for the fan. As it is difficult to arrange for recirculation, thermal efficiencies are low, and it would appear that an overall efficiency of 50% represents reasonable working conditions.

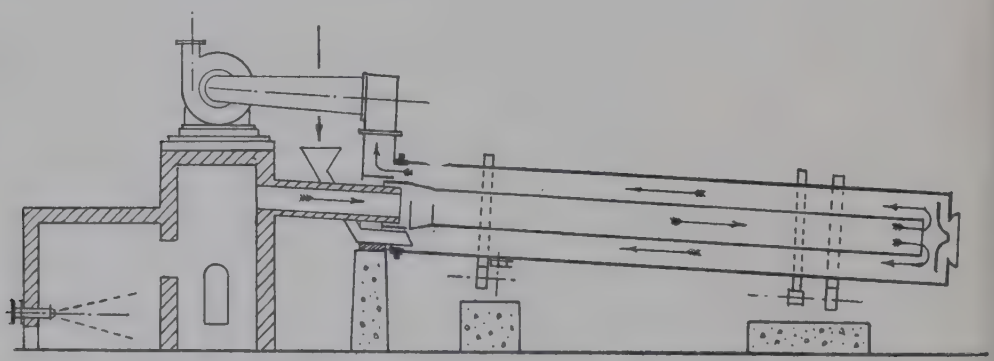


Fig. VII–10.—High-Temperature Double-Shell Dryer.

The entrainment of solid in the air-stream, which is a drawback in rotary dryers, is turned to advantage in pneumatic-conveyor dryers. These units, as shown in Fig. VII–11, use high gas velocities to entrain all the material being dried. A rotating valve system throws the solid particles into a rising gas main, and the hot gas is given a sufficient velocity to lift them bodily into a cyclone separator or "expansion chamber" where separation can occur. Heat exchange is claimed to be very rapid, and undoubtedly a high degree

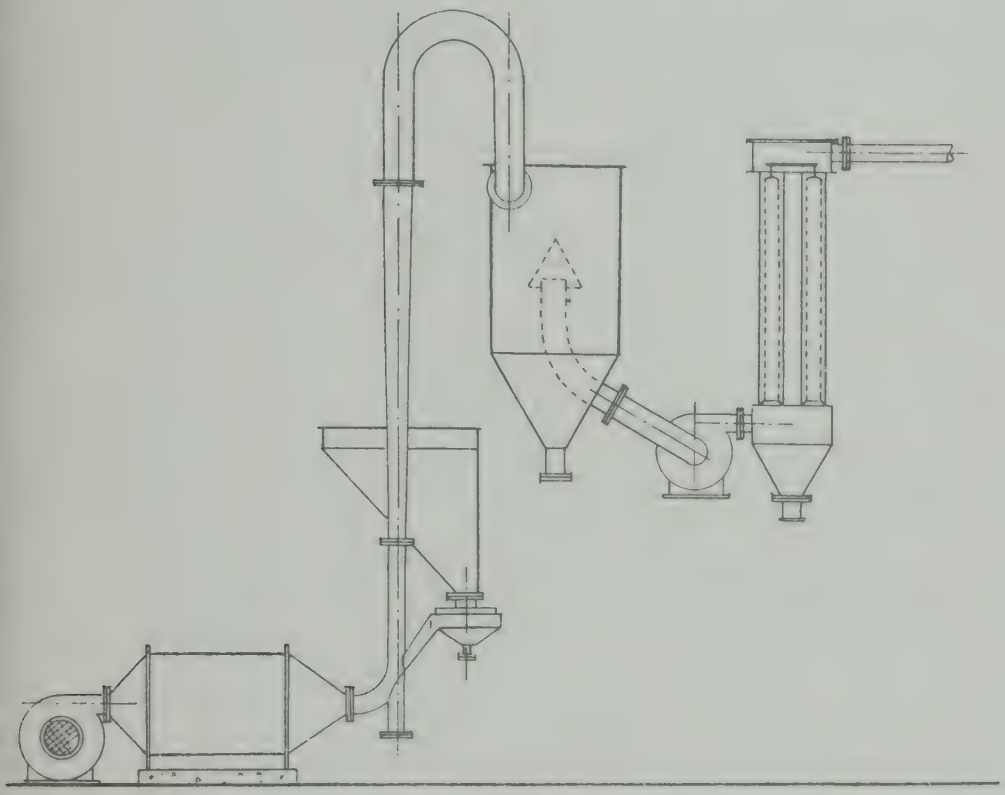


Fig. VII–11.—Pneumatic-Conveyor Dryer.

of turbulence obtains immediately around the solid-entry section. When all the moisture to be removed is on the solid surface, drying may be rapid, but where this moisture has to come from the interior of the solid particle, the short time of drying will probably leave much moisture behind. This is recognised in recent developments, which embody a long rising section in which the solid particles are carried upwards only slowly, after a rapid dispersion. Apart from this the main drawback of these conveyor dryers is the high power requirement for air circulation. It is often the case, however, that the wet solid must be lifted in any event, and then the

pneumatic conveying can be combined with drying. In a typical instance ammonium sulphate crystals from a centrifuge were dried from 4% moisture down to 0·5% while being lifted to a hopper 20 ft. above the centrifugal discharge. The conveyor dryer is the first of a series of drying units in which the total contact time with the drying gas is measured in seconds.

This short contact time may be very important with thermally sensitive substances, and is the basis of the design of roller film dryers. These dryers take the concentrated solution, or a solid–liquid suspension, as feed, and convert this to a near dry solid in a

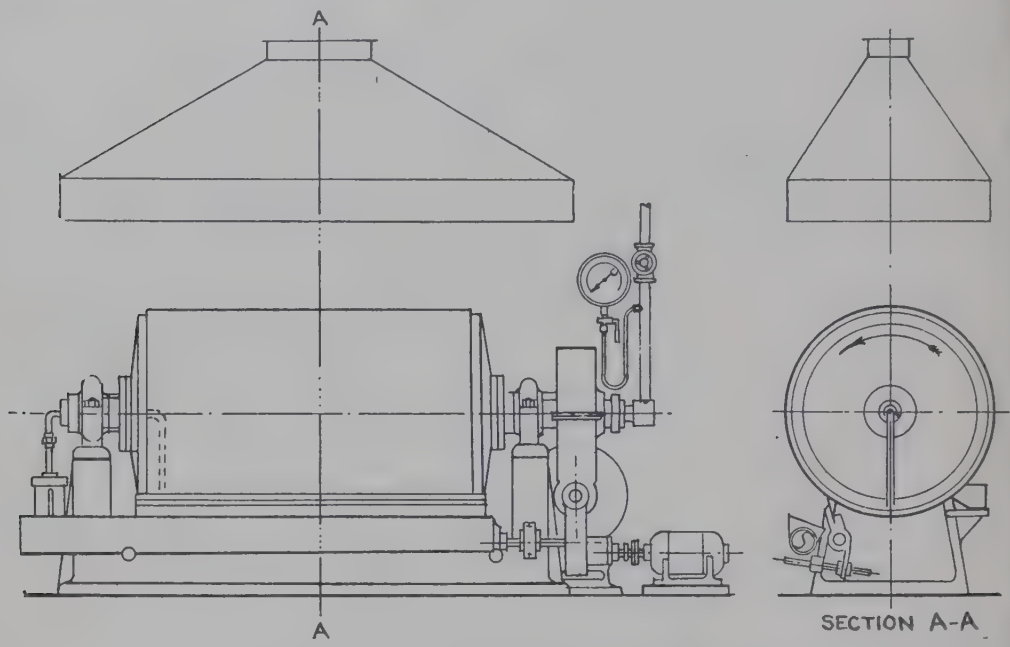


Fig. VII–12.—Roller Film Dryer.

minute or less. The film dryer is made up from a hollow steam-heated roller, to which the feed is applied evenly at one place as the roller turns; the film is conveyed by the roller, drying as it goes, and by the time a revolution has been almost completed is dry enough to form a solid flake, easily detached from the roller by a scraper knife. A general arrangement drawing for a single-roll dryer is shown in Fig. VII–12; the solution is spread on the roll by a side-feeding trough, which can be adjusted to give a varying depth of " pick-up ". This feed system is the weak point of the roller dryer, as it is essential that the film thickness shall be even. An uneven film bakes hard in some places and is almost fluid in others when it approaches the discharge knives, and the resulting uneven discharge

makes for a still more uneven distribution in the next revolution. To overcome this difficulty a number of feed systems have been used, including a subsidiary feed roller immersed in the liquid and unheated, and a series of sprays. It would appear that the most successful method is to build the drying rolls in pairs, as shown in Fig. VII–13. These rollers are held against each other by springs, and the feed liquid is contained in the trough formed between them. Then as the rollers turn in towards each other they convey an even film down through the clearance slit. This is dried and discharged by scrapers as shown in the diagram; it is notable that any failure in removal results in dry-solid cakes forcing the rollers apart, with

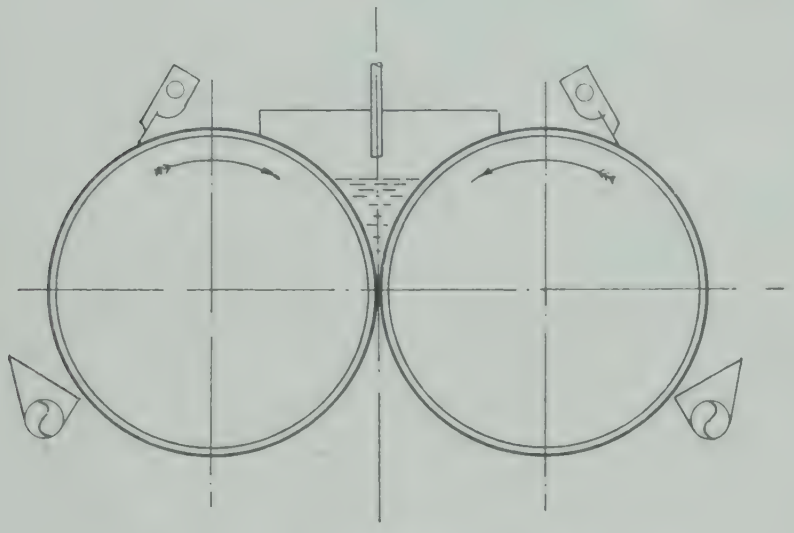


FIG. VII–13.—DOUBLE-ROLL FILM DRYER.

unfortunate results to the dry-solid receiver below. As a result of these troubles it is usually necessary to have careful watch kept on these dryers, and the labour charge, apparently negligible, is really rather high unless the drying rolls are large—say 2 ft. 6 in. in diameter by 6 ft. long. Such a roller dryer will take 3 h.p. for driving at about 10 r.p.m., and will evaporate up to 4 lb. of water/sq. ft./hr. from saturated solutions. On a basis of 50% solids in the feed this means a similar output of dry solid. It is usual to arrange for the solid to be discharged with about 5% of moisture still remaining; such cake is much more easily discharged than " bone-dry " material. The cake removal is also improved by using highly polished rolls of stainless steel or chromium-plated mild steel. The low heat transfer through the stainless steel is something of a disadvantage, as

heat-transfer rates of the order of 200–300 B.Th.U./sq. ft./hr./° F. have been claimed for these dryers, and have, in fact, been suggested as a basis for design. Finality has not been reached in the design of these roller dryers, owing to the difficulties of application and removal of the film, but the very short drying time—about 5 sec. at 10 r.p.m.—will always make them attractive for thermally sensitive materials. There is no difficulty in enclosing the whole unit in an airtight casing and carrying out the drying under reduced pressure, though the dried film approaches the temperature of the hot surface at the end of the drying cycle.

Still more rapid drying can be obtained by the use of spray drying units. A concentrated solution or suspension is sprayed into a large chamber—not less than 10 ft. in diameter as a rule—in which the liquid spray meets a current of drying air. This current may travel either with or against the liquid droplets, vaporising the water and giving a dry powder, which drops to the bottom of the chamber or is conveyed to a cyclone collector. As a rule the hot gases are formed by burning oil or fuel gas in a separate furnace chamber and then mixing with excess air to give the inlet air temperature required. As with all direct dryers, the higher the inlet air temperature the higher the thermal efficiency which can be attained without reheating (Fig. VII–6). Even though the material is thermally sensitive, it is possible, as shown in Fig. VII–14, for the wet material to come into contact with the hot gases, and for the partly dried particles to meet the cooler section of the gas-stream. In the initial drying stages the droplets can only heat up to the wet-bulb temperature of the air-stream, while the rapid drying cools the hot gas very rapidly. The whole period of drying is only about 3 sec., which produces some interesting physical effects in the dried particles. It has been shown that certain products are in the form of hollow spheres, the size of which can be regulated by methods of spraying. The most popular type of spray appears to be a small rotating disc, spinning at rates from 5000–20,000, r.p.m. As this speed increases under steady drying conditions, the product changes from solid droplets to hollow spheres, which gradually increase in size until they disintegrate to shell fragments at the highest speeds.* Many of the physical characteristics of the solution persist in the dried particles; it has been suggested that dried emulsions give particles with the external phase persisting as a

* Phillip, *Trans. Inst. Chem. Eng.*, 1935, 13, 116.

layer on the solid, while vat dyestuffs dry to spheres coated with a film of dispersing agent.

It is essential that the drying be completed before any of the solid touches the walls of the chamber, or a deposit will rapidly build up. The air is therefore given a definite rotary swirl by passing it through tangential entry ports: there may be two sets of these parts, giving an outer and an inner zone revolving in opposite directions. An additional precaution is to build the chamber with highly

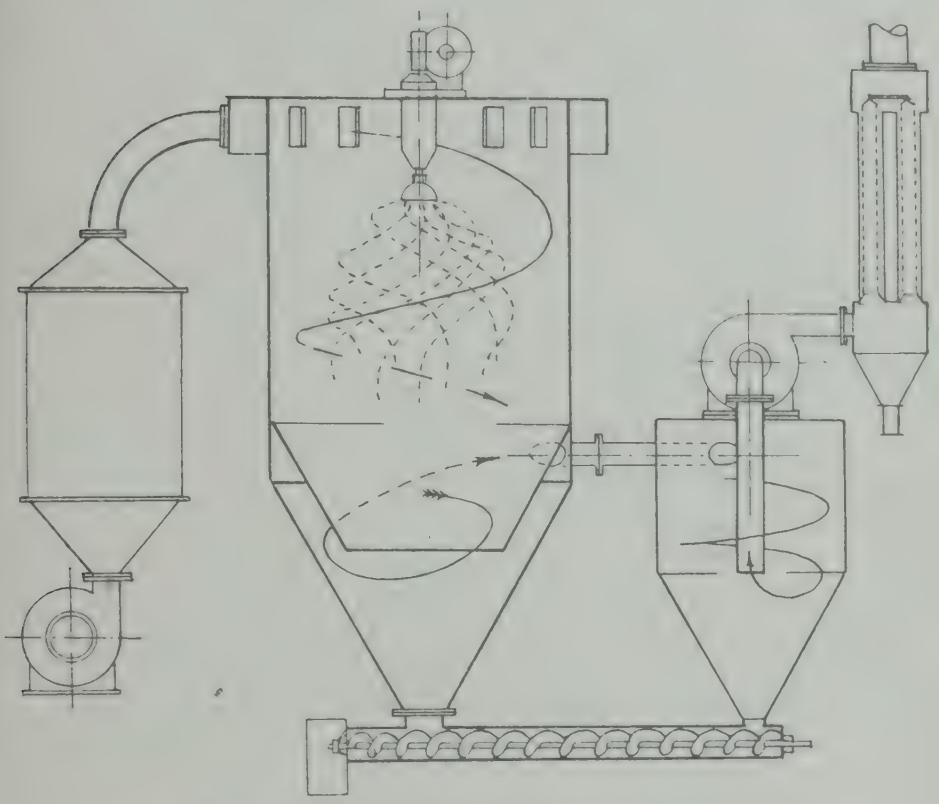


FIG. VII–14.—CO-CURRENT SPRAY DRYER.

polished walls of tin or chromium plate, and it has been suggested that the ideal wall would be made from "Teflon", an extremely inert plastic material to which no material will adhere. The solid is always partly entrained in the air leaving the chamber, and it is necessary to keep the outlet air temperature well above the dew-point. The exit gases can then be passed through a cyclone precipitator or a bag filter battery, as shown in Figs. VII 14, 15. Between 10% and 20% of the total solid yield is recovered in this way. It will be seen from a study of the two systems shown in Figs. VII 14 and VII 15 that in the zone immediately surrounding the spray the

solid and gas are travelling (14) co-current and (15) counter-current. It is probable that slightly higher thermal economies can be obtained with the latter, while thermally sensitive substances are best treated by the co-current process. In any case a large proportion of the powder flows co-currently with the gas, as indicated above. The thermal efficiency cannot be high in view of the impossibility of reheating and of the high outlet temperatures and is usually about 40% of theory.* It may be slightly improved by passing the outlet gases through a scrubbing tower down which the feed solution

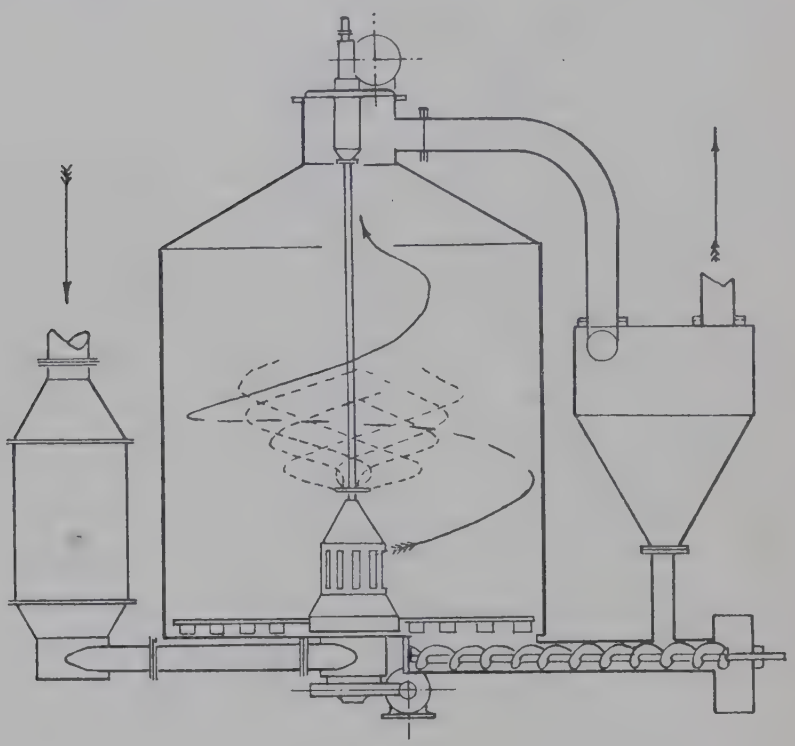


FIG. VII–15.—COUNTER-CURRENT SPRAY–DRYER.

is flowing; the solution is preheated to some extent, while the gas is finally stripped of residual solids. It would appear that the rate of heat transfer from the hot gas to the material being dried is from 3–5 B.Th.U./cu. ft./hr./° F., but actual experimental data are scanty. In a typical unit of the co-current type, a vegetable extract could be dried from a 45% solution to a solid containing 5% water at a rate of 350 lb. of dried solid per hour. The drying air entered at 200° C. and left at 90° C., while the solution-solid seemed to remain

* *B.I.O.S. Report* 1587.

at about 60° C. throughout. The dryer chamber was a cylinder, 11 ft. in diameter by 11 ft. high, with a coned base adding another 9 ft. to the height.

It has recently been noted that the process of drying painted surfaces can be notably hastened by radiant heat treatment. Clearly the most rapid way of passing heat to a good absorbing surface is by radiation, and it is probable that a good proportion of the heating in any indirect dryer is by this means. Dryers are now constructed with the special intention of making use of this radiated heat—the

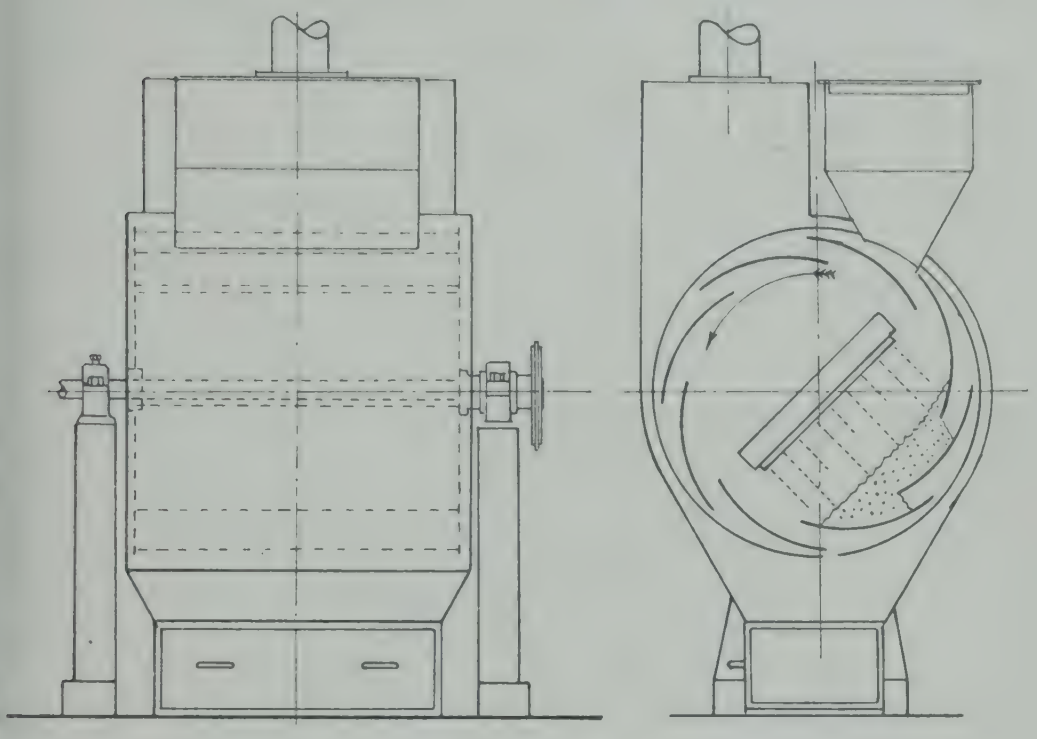


Fig. VII–16.—Infra-Red Radiation Dryer.

so-called "infra-red" units. Their use is highly specialised, and in view of the high cost of such radiated heat it is probable that they will only be used when other considerations than drying cost enter into the process. The radiation can either be from low-temperature electric lamps or gas-heated panels; one of the latter type is shown in Fig. VII–16. A rotating drum carries the drying solid, and a radiating panel fixed at the centre heats the powder surfaces as the solid particles tumble. It may be possible to adjust the wavelength of the radiation to heat certain materials preferentially, and the rapid hardening of paint films has been traced to some such effect. For

ordinary chemicals it is probable that the method will prove too expensive, but the matter has not been settled.*

The same considerations will probably apply to the use of radio-frequency dielectric heating in drying problems. It is practically possible to boil out moisture from the interior of a porous solid without heating the outside in any way, but all this heat has to be supplied in the form of electrical energy at very high frequencies. This charge has been estimated at four times the initial cost of the electricity as originally supplied in normal form.† and as a result the principle will only be used for very special drying problems. It has been suggested, for instance, that penicillin can be very rapidly dried *in vacuo* without any contact with heating agents such as the hot wall of a containing flask.

A more general but still costly technique which has lately been applied in the drying of thermally sensitive substances is the process of freeze-drying. The material—usually a solution—which is to be treated in this way is first frozen, and the ice is then sublimed off in vacuum of the order of 100–300 microns at temperatures between — 10 and — 30° C. The process has been outstandingly successful in dealing with such materials as blood plasma and penicillin, and produces unique dried solid masses in that the original structure of the frozen solid persists throughout, leaving a honeycomb structure through which the water molecules move readily during the drying period. The process calls for exact control of heating, so that the ice is removed at the highest possible rate consistent with continued freezing. The ice " vapour " is usually condensed, again as ice, on a refrigerated condensing surface, from which it is removed by scraping, a fairly easy operation, as the water condenses as " hoar frost ". It is possible, of course, to pump off the water vapour and discharge it through the pumping system to atmosphere, but this calls for the use of multi-stage ejectors, and it seems economic to condense in the vacuum system. The design of freeze-drying units is in its infancy, but it appears to be possible to remove ice from the surface of a solid cake at a rate of 1 mm. per hour. This corresponds to a rate of evaporation of 0·2 lb./sq. ft./hr., so that rates are commensurate with those obtained from normal vacuum-oven work. At the same time the very exhaustive evacuation will call for

* See, e.g., Reavell, *Trans. Inst. Chem. Eng.*, 1944, 22, 13 ; Tiller and Garber, *Ind. Eng. Chem.*, 1942, 34, 773.

† Stephens, *Trans. Inst. Chem. Eng.*, 1949.

carefully made plant and expensive pumping units, and it has been suggested that the cost of water removal will certainly not be less than 3*d*. per pound (1948). The method is still novel, and very few working figures are available.* The only large plant used outside the medicinal and pharmaceutical fields would appear to be the one described by Schwarz and Penn,† which handles 60,000 gallons of orange juice per day. The conditions of drying in this plant are reminiscent of vacuum roller drying, as it is doubtful if the solution ever freezes. The temperature is low, and the water is condensed as ice on a refrigerated roller condenser.

* H. H. Chambers, *Trans. Inst. Chem. Eng.*, 1949.
† *Ind. Eng. Chem.*, 1948, 40, 938.

CHAPTER VIII

MIXING

The object of mixing in chemical processes is to ensure that a nearly homogeneous mixture is formed and maintained with as much completeness and as low a power input as possible. This is true for all processes, whether the ultimate goal is an increase in the rate of heat transfer, of chemical reaction, or of mass transfer. The diversity of purpose for which mixing may be required makes it very difficult to formulate or measure any definite criterion of mixing efficiency, although the power input can be determined fairly accurately. The degree of fluid turbulence obtained (assuming all mixing as being between fluid phases) is probably the best measure of mixing, but varies very widely in different parts of a mixing vessel and can only be measured with difficulty. The usual measures of mixing are indirect functions, such as rate of heat transfer or rate of solution of a soluble solid. These may involve a variety of confusing elements; in a typical case, heat transfer to stirred liquid is proportional to power input and stirrer speed up to a point, but aeration of the liquid then destroys the relationship completely. Again, the rate of nitration of an aromatic hydrocarbon by mixed acids is proportional to stirrer speed for poor agitation, but is quite independent of agitation if the mixing is good. A symposium on mixing held in 1944 concluded that no suitable general criterion of mixing efficiency existed.*

The typical mixing device is some form of solid surface, which is moved rapidly through a fluid mass. The frictional force F, caused by a relative velocity v in a fluid of density ρ, can be expressed in terms of a Reynolds Number, as in the equation

$$\frac{F}{\rho v^2 D^2} = f\left(\frac{vD\rho}{\eta}\right) \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

Here D is a characteristic dimension of the surface, and η is the fluid viscosity. If the surface is rotating at a speed N, the velocity

* *Ind. Eng. Chem.*, 1944, 36, 488–522.

c becomes $IIDN$. As the power required (P) is a product of force and velocity, a general equation may be written

$$\frac{P}{\rho N^3 D^5} = f\left(\frac{ND^2\rho}{\eta}\right) \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

By analogy with ordinary fluid flow, it would be expected that plots of $P/\rho N^3 D^5$ against $ND^2\rho/\eta$ would show viscous and turbulent flow regions. This has, on the whole, been supported by experimental evidence,* but the matter is in some dispute; the measure of D is not simply a question of diameter of paddle or propellor, as suggested in the argument above; but must take into account the dimensions of the mixing vessel and other factors. In fact, two schools of thought have developed, one of which uses the agitator diameter in correlative equations, while the other prefers a dimension based on the mixing vessel. As a result a number of modifications of equation (2) above have been suggested, all somewhat limited in their application. For instance, Stoops and Lovell† propose that, for a propeller stirrer of diameter D_p in a tank of diameter D_t, results may be correlated by the relation

$$\frac{P}{\rho N^3 D_p^5} = 0{\cdot}56\left(\frac{\rho N D_p^2}{\eta}\right)^{0{\cdot}81} \times \left(\frac{D_t}{D_p}\right)^{0{\cdot}93}$$

Hixson and Baum,‡ using a turbine mixer with a diameter $\frac{1}{3}$ that of the mixing tank of diameter D_t, gave as the correlating equation

$$\frac{P}{\rho^{0{\cdot}88} N^{2{\cdot}88} D_t^{4{\cdot}76}} = 1{\cdot}5\eta^{0{\cdot}12} \times 10^{-12}$$

for all values of $\left(\frac{\rho N D_t^2}{\eta}\right)$ above 10^4. It would appear that, as a general rule, the power requirements will vary as a function of $\rho N^3 D^5 \eta^{0{\cdot}2}$, but this formula can only be applied within narrow limits for the scaling-up of figures obtained on similar plants.

These equations give a measure of power consumption but not of mixing efficiency. The type of mixing device used in any particular case must be determined by experiment, particularly with fluid systems which exhibit thixotropic tendencies. In such cases

* *Ind. Eng. Chem.*, 1942, 34, 120 and 194 (corrected formula).
† *Ind. Eng. Chem.*, 1943, 35, 845.
‡ Hixson and Baum, *loc. cit.*

it is necessary to ensure that the rate of shear produced by a mixing device does not fall below a certain critical value in any part of the mixing vessel, or the fluid in that neighbourhood will cease to move. All, or nearly all, suspensions of solids in liquid can exhibit such thixotropic behaviour. In addition to this, a mixture of two liquids can show viscosities very far removed from those of the components when a stable dispersion is formed of one in the other.*

The simplest and oldest device for mixing liquids or slurries is the paddle mixer, with two or more arms or a frame mounted on a

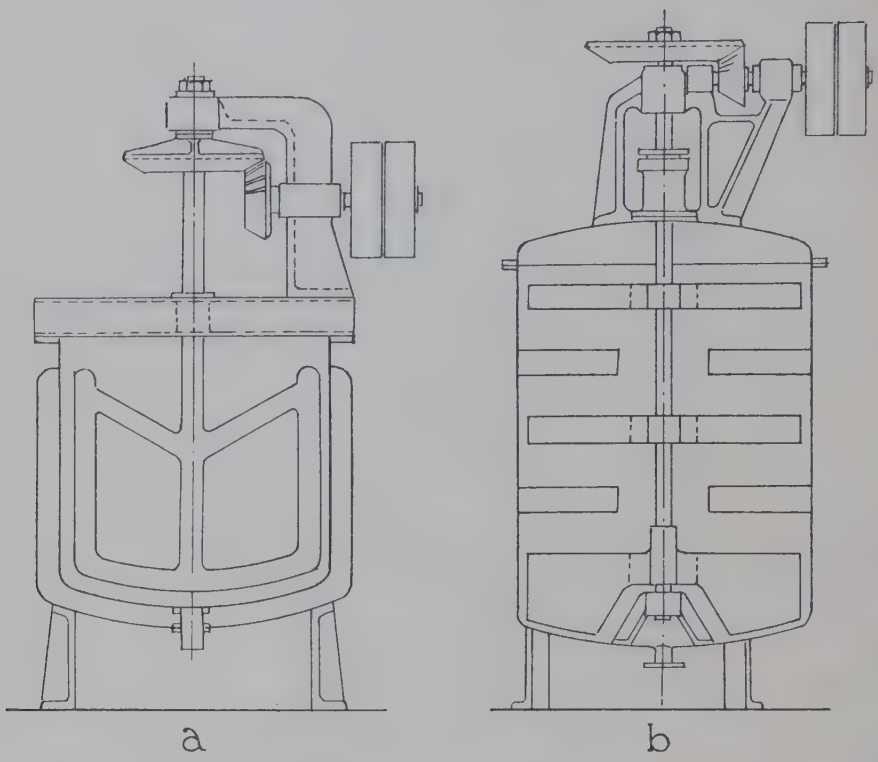


Fig. VIII–1.—Paddle Mixers.

central shaft and rotating at a speed of 20–100 r.p.m. A typical paddle stirrer of this type is shown in Fig. VIII–1a, with arms sweeping the surface of the vessel fairly closely and leaving very little space unswept by the mixing arms. By the use of an interlocking arrangement of baffles, as shown in Fig. VIII–1b, it is possible to ensure that all areas of the vessel are within a few inches of a stirrer. This means that the paddle device can be employed with advantage in mixing viscous or thixotropic liquids. For simple paddles made of two flat blades set at an angle of 45° to the

* Miller and Mann, *Trans. Amer. Inst. Chem. Eng.*, 1944, 40, 709.

vertical and 90° to each other, the power requirements (P = horsepower) have been given by White and Brenner* as

$$P = (1{\cdot}29 \times 10^{-4}) N^{2{\cdot}86} D_p^{2{\cdot}72} W^{0{\cdot}3} D_t^{1{\cdot}1} H^{0{\cdot}6} \rho^{0{\cdot}86} \eta^{0{\cdot}14}$$

where W = the paddle width, H = the liquid height, and all other terms are expressed in F.P.S. units.

Example 1. *A simple two-blade paddle stirrer, 5 ft. in diam., with blades 6 in. wide, is to revolve at 30 r.p.m. in a 6-ft. diam. tank, with a working liquid depth of 4 ft. The liquids being mixed have the viscosity and density of water at 20° C.*

Then power required

$$= (1{\cdot}29 \times 10^{-4}) \times (0{\cdot}5)^{2{\cdot}86} \times (5)^{2{\cdot}72} \times (0{\cdot}5)^{0{\cdot}3} \times (6)^{1{\cdot}1} \times (4)^{0{\cdot}6} \times (62{\cdot}3)^{0{\cdot}86} \times (6{\cdot}72 \times 10^{-4})^{0{\cdot}14}$$

$$= (1{\cdot}29 \times 10^{-4})(0{\cdot}138)(79{\cdot}5)(0{\cdot}813)(7{\cdot}18)(2{\cdot}24)(34{\cdot}8)(0{\cdot}36)$$

$$= 0{\cdot}23 \textit{ h.p.}$$

The formula has been criticised by later workers but appears to give reasonable results. Some general rules for paddle-mixing, suggested by Asquith† from a study of published results, are given below:

(1) Paddle-tip velocities between 300 and 400 ft./min.
(2) Paddle located near bottom of vessel.
(3) Paddle length just greater than vessel radius.
(4) No baffles required.
(5) Ratio of vessel diam. to liquid depth "reasonably near" to 1.

When baffles are introduced, the rate of mixing and the power consumption are both increased, and evidence as to advantage is dubious, except with very viscous liquids.

It is probable that paddle mixers are dropping out of use in favour of higher-speed mixing devices, except for liquids with viscosities of above 0·5 poises and for light solid suspensions with a marked tendency to form "structures" in the mixing tank. Even in these cases devices such as an air-lift agitator may be substituted for the paddle. In this agitator two sets of arms are mounted on a central hollow shaft, as shown in Fig. VIII–2a. The lower set move

* *Trans. Amer. Inst. Chem. Eng.*, 1934, 30, 585.
† *Trans. Inst. Chem. Eng.*, 1945, 23, 10.

slowly over the flat base of a cylindrical mixing tank, and carry the settled solids towards the tank centre. Here an air lift conveys the thickened slurry continually up the central shaft, to flow from ports just above the liquid surface into the upper arms. These are rotating launders, or perforated fluid conduits, which shower the lifted material over the surface of the main suspension. As the rate of rotation is low, and no attempt is made to set the fluid in rapid motion, power requirements are small, a tank 30 ft. in diameter

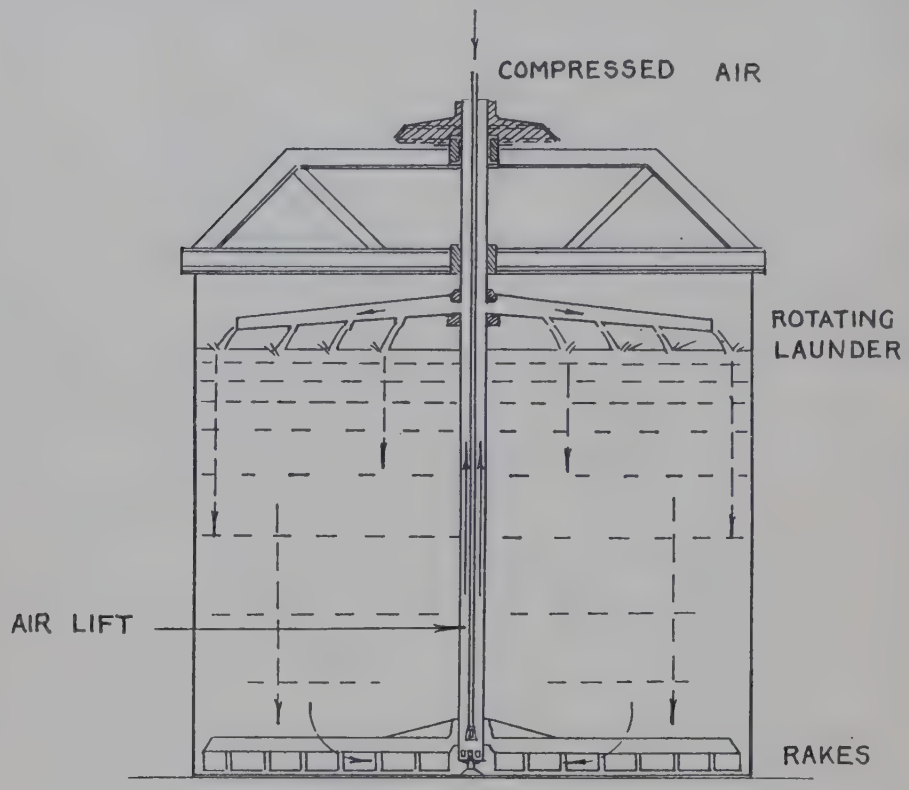


Fig. VIII–2a.—Air-lift Agitator.

by 15 ft. deep taking only 2 h.p. to drive rakes and launders at 2 r.p.m. The air required for the air lift is, of course, an extra charge on power, but for the same tank is only 30 cu. ft./min., representing another 2 h.p. The total power requirements for large vessels of this form can be as low as 0·4 h.p. per 1000 cu. ft. of stirred volume. Where air is not desirable, a larger central shaft can carry an alternative lifting device (Fig. VIII–2b).

Turbine mixers are increasing in popularity, especially for forming and sustaining suspensions of heavy solids, and for the dispersion of fluids of very different densities. The normal turbine is a rotor

made from a flat disc set on a central hub, with short vertical blades arranged either radially or curving backwards from the centre

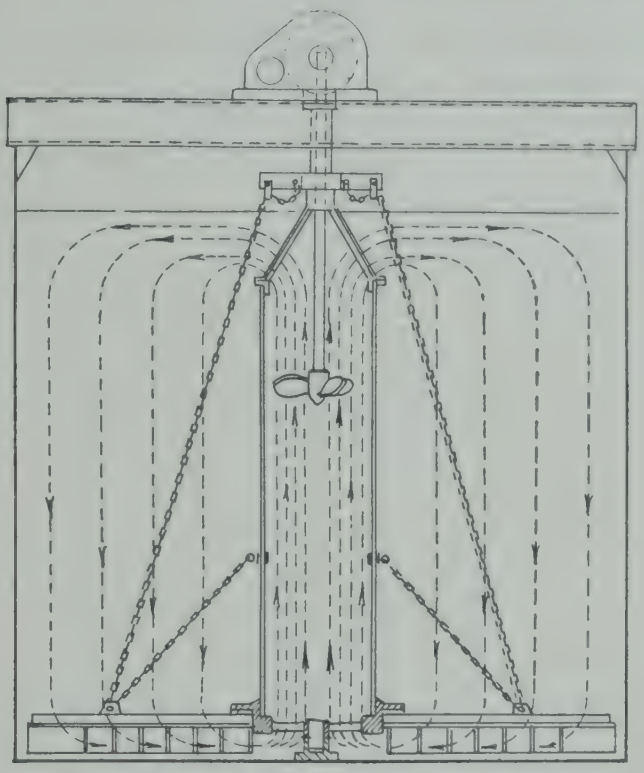


Fig. VIII–2b.—Slurry Agitator.

of rotation. This latter arrangement shows the intimate connection with the liquid centrifugal pump; the turbine mixer is in fact a

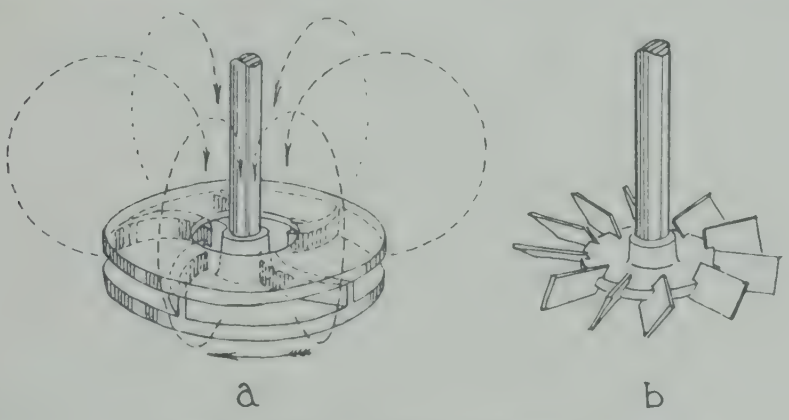


Fig. VIII–3.—Turbine Mixers.

glandless pump, sucking in liquid at the centre and discharging it at the periphery, as shown in Fig. VIII 3a. An alternative form (Fig. VIII 3b) dispenses with the central disc, and approximates to a

multi-blade propeller. This gives a positive one-way flow through the blades and better mixing in a vertical direction, but may leave outer layers relatively undisturbed. It is probable that 3a will be better for heat transfer and 3b for reaction between a liquid and a solid in suspension.

The introduction of vertical wall baffles, very common with this form of mixer, makes for much more efficient mixing, but increases the power consumption very considerably—up to four times the power required in an unbaffled tank. The power increases with the number of vertical side baffles up to six, but further baffling appears to have little effect.* It is not known if all this increase in power is absorbed in more efficient mixing, but the appearance of the fluid mass certainly suggests a great improvement. In particular the deep vortex formed by a simple central turbine disappears, and air entrainment, which may be undesirable, is almost eliminated. These vertical side baffles should always be used when the liquid is being heated or cooled by a coil, but can be replaced by a stator ring round the turbo-impeller if only mixing effects are required.† The actual bladed disc is made in sizes ranging from 6 in. to 3 ft., and as a rule the ratio of tank to turbine diameter is between 4 and 6. The equation for power consumption already quoted gives the horse-power as

$$P \text{ (horse-power)} = 1{\cdot}5 \times 10^{-12}\eta^{0{\cdot}12}\rho^{0{\cdot}88}N^{2{\cdot}88}D_t^{4{\cdot}76}$$

where η, ρ, N, and D_t are in C.G.S. units.

Example 2. *A turbine agitator, 9 in. in diam., in a 3-ft. tank, is stirring at* 300 *r.p.m. a liquid of density* 1·1 *and viscosity* 0·02 *poises. Then the power required is given by*

$$P = 1{\cdot}5 \times 10^{-12} \times (0{\cdot}02)^{0{\cdot}12} \times (1{\cdot}1)^{0{\cdot}88} \times (5)^{2{\cdot}88} \times (91)^{4{\cdot}76}$$

$$= 1{\cdot}5 \times 10^{-12} \times 0{\cdot}626 \times 1{\cdot}09 \times (1{\cdot}025 \times 10^2) \times (2{\cdot}113 \times 10^9)$$

$$= 0{\cdot}22 \text{ h.p.}$$

A large number of proprietary makes of stirrer can be classed along with turbine mixers, but are somewhere between the paddle type and the true centrifugal turbine design. The " Typhoon " agitator shown in Fig. VIII–4a may be regarded as a two-bladed centrifugal.

* Mack and Kroll, *Chem. Eng. Prog.*, 1948, 44, 189.
† Bissell *et al.*, *ibid.*, 1947, 43, 649.

and produces the same mixing patterns as the unit shown in Fig. VIII–3a. It is combined with top baffles to destroy the vortex, a device which has been widely used with all central high-speed stirrers. Fig. VIII–4b shows a " bucket " agitator, used in reaction vessels to draw a steady stream of liquid over cooling coils. It may be mounted in a central guide tube, and has proved very successful in mixing reactors for organic nitration. Another form of mixer which is claimed to be particularly suitable for viscous liquids is

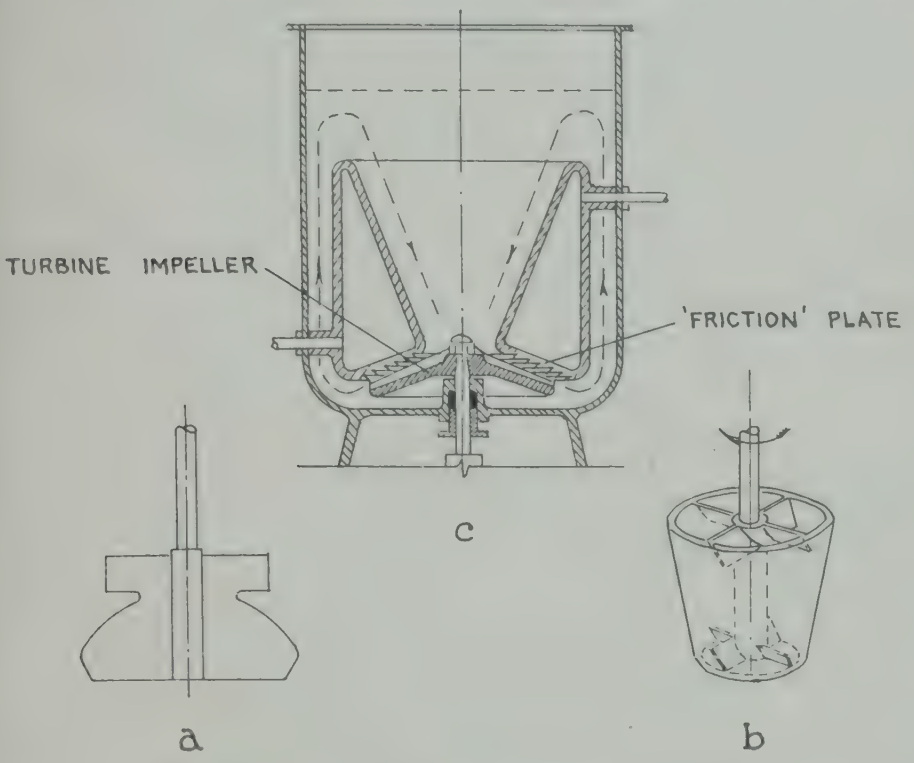


Fig. VIII–4.—Typical Specialised Agitators.
(Courtesy of Lang-London, Ltd. Christie and Gray, Ltd.)

shown in Fig. VIII–4c, and is built up from a turbine impeller set close to a " friction plate " baffle. A powerful local shearing action is set up, and the mixer is designed to homogenise batches of liquid of the consistency of cream. It can be built in sizes from 1 gallon (speed up to 3500 r.p.m.) to 1100 gallons (speed 400 r.p.m.).

Only such very high-speed units as the small mixer mentioned above can be driven by direct coupling to an electric motor. It is doubtful whether this is good practice, because of the difficulty of dismantling for repairs, and the general method is to drive through crown-wheel gearing and a multi-vee belt drive. Individual electric

motors, mounted in any convenient position, are now almost invariably used for chemical plant units. An exception is found in the explosives industry and in similarly dangerous situations, where all electric motors must be outside the working buildings. Shafting must be used here, and fairly slow turbo-mixers or paddles are more suited to this type of drive than are the higher-speed propeller mixers.

These high-speed propeller stirrers are the simplest of all mixing devices, and can be arranged in many ways. The portable stirrer shown in Fig. VIII–5a is directly coupled to the shaft of an electric

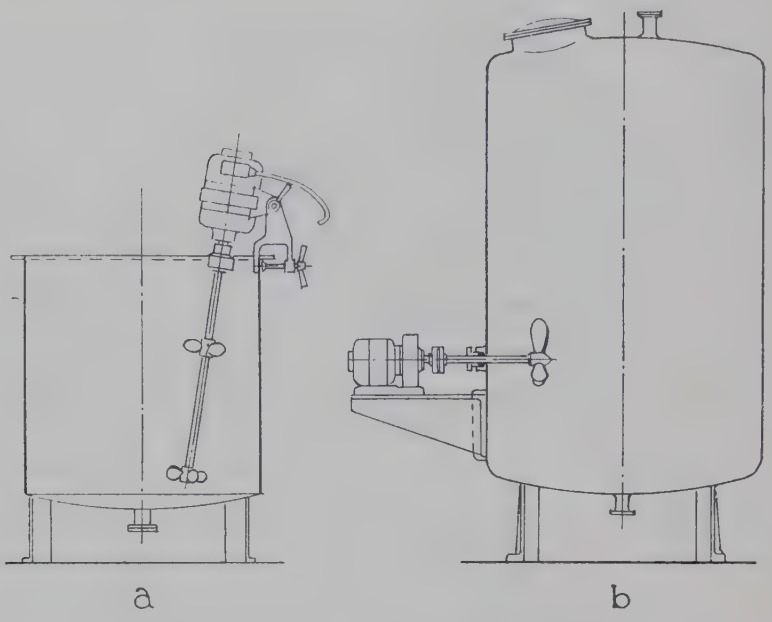


Fig. VIII–5.—Propeller Mixers.

motor with reinforced ball bearings. It can be clamped on to the side of a reaction vessel for a short working period, removed, and used for another reaction. The side eccentric mounting gives thorough agitation and avoidance of swirl without the aid of baffles. The high speed (anything up to 1500 r.p.m.) and small impeller size make the mixing almost useless for viscous or thixotropic fluids. Unsupported shafts revolving at these speeds should be as short as possible, and for large tanks it is usual to mount two or more propellers as side-entering agitators, as in Fig. VIII–5b. Although normally the propeller is only used for mixing mobile liquids, it has been claimed that mounting in a draft tube gives a high local rate of shear which is suitable for making emulsions.

The propeller stirrer, mounted centrally, will give a vortex similar to that obtained with the turbine, and some baffling will be necessary to check this effect. The small size of the impeller is an advantage when stirring has to be carried out in a restricted space, such as the cone bottom of a tank from which a mixture is running under continued agitation, but the propeller should be covered by a liquid " seal " of at least 1·5 propeller diameters if it is to work efficiently.* A formula for the power requirements of a stirrer given by Stoops and Lovell† is

$$P = 0{\cdot}56\rho^{0{\cdot}8}\eta^{0{\cdot}2}N^{2{\cdot}8}D_p^{3{\cdot}7}D_t^{0{\cdot}6} \times 1{\cdot}82 \times 10^{-3}$$

(The original formula is for consistent units, and the factor $(1{\cdot}82 \times 10^{-3})$ converts the power figure from F.P.S. to h.p.)

Example 3. *A propeller stirrer, 6 in. in diam., is stirring in a 3-ft. diam. tank a liquid of density 68·5 lb./cu. ft. and viscosity 0·02 poises* $(1{\cdot}34 \times 10^{-3}$ *F.P.S. units). The stirrer speed is 600 r.p.m. The power required is given by*

$$P = 0{\cdot}56\ (68{\cdot}5)^{0{\cdot}8} \times (1{\cdot}34 \times 10^{-3})^{0{\cdot}2} \times (10)^{2{\cdot}8} \times (0{\cdot}5)^{3{\cdot}7} \times (3)^{0{\cdot}9} \times 1{\cdot}82 \times 10^{-3}$$

$$= 0{\cdot}56 \times 29{\cdot}4 \times 0{\cdot}266 \times 631 \times 7{\cdot}58 \times 10^{-2} \times 2{\cdot}69 \times 1{\cdot}82 \times 10^{-3}$$

$$= 1{\cdot}02\ h.p.$$

The use of agitation with a high local shear rate has been indicated above as necessary to make a stable emulsion, or dispersion of one liquid phase in another. Most reactions between two liquids, such as nitration or washing, require only a coarse dispersion with droplets of the dispersed phase as large as 0·1 mm. in diameter. Stable emulsions require a drop size less than 0·01 mm. and generally around 0·0015 mm. Various specialised mixing devices have been devised to give the high local stresses required to produce these very fine droplets, and the machines are usually supplied with an ordinary liquid mixture as feed. The " colloid mill " shown in Fig. VIII–6 has a high-speed cone-shaped rotor pressed closely against a matching stationary surface. As the rotor moves, at speeds of 10,000 r.p.m. for a 10-in. rotor, the centrifugal forces set up draw the mixture contained in a top reservoir down through the narrow

* Bissell *et al., Chem. Met. Eng.*, 1946, 53 (No. 1), 118.
† *Ind. Eng. Chem.*, 1943, 35, 845.

gap, which is maintained at about 0·005 mm. clearance. In a typical case tar oil and water containing 0·02% of dispersing agent were mixed by ordinary stirring and fed through the colloid mill to give a dispersion of oil in water which contained no droplets larger than 0·002 mm. An alternative to the colloid mill is the jet homogeniser, in which the two liquids are forced through a small hole under pressure. In the Weir homogeniser a triplex ram pump delivers the mixture under pressures up to 5000 lb./sq. in. to a carefully ground spring-loaded release valve. The spring is set to lift sufficiently to give the pressures required for dispersion.

The mixing of very viscous fluids, with viscosities of above 20 poises, can be carried out with paddle mixers combined with fixed

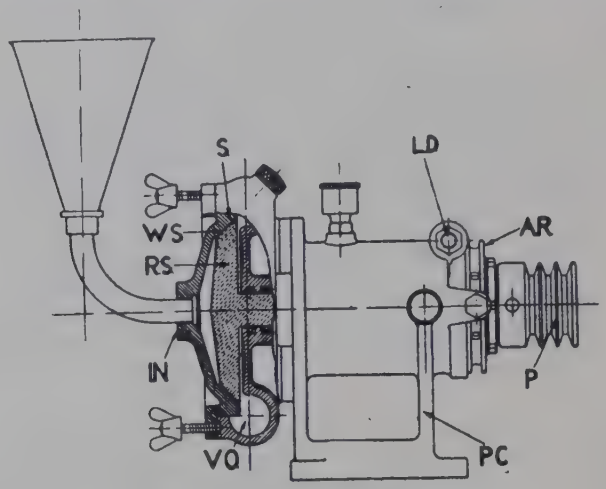


Fig. VIII–6.—Colloid Mill.
(*Courtesy of Premier Colloid Mills, Ltd.*)

baffles. The power requirements and the stresses on the stirrer arms and baffles are very heavy, and the plant becomes very heavy and clumsy. For a mixture such as rubber cement, a paddle baffle arrangement such as that shown in Fig. VIII–7 has been used. The clearances are small, and the scraping action of the blades makes it possible to heat or cool through the walls, though heat-transfer rates must be low. A 200-gallon vessel of this type took 7 h.p. to drive the stirrer at 20 r.p.m. As the mixture to be handled passes to the consistency of a bread dough, kneading pans are used. These are short U-section vessels, with kneading arms of irregular spiral section rotating at 10 r.p.m. Even at this slow speed, motors of 10 h.p. are required to turn the kneaders in a 100-gallon pan. It is claimed that the setting of these arms with regard to each other

and the sides of the vessel has a great effect on the power consumption and the amount of heat developed. As the heating-up in a charge of low heat capacity can be considerable, special water-cooled pans have been employed, both in the baking and in the rubber industries. These kneading pans may be replacing the mixing rolls, peculiar to the rubber industry, which spread out the mix continually on two rollers, as in a roller mill.

An alternative form of mixer for pastes which must be squeezed

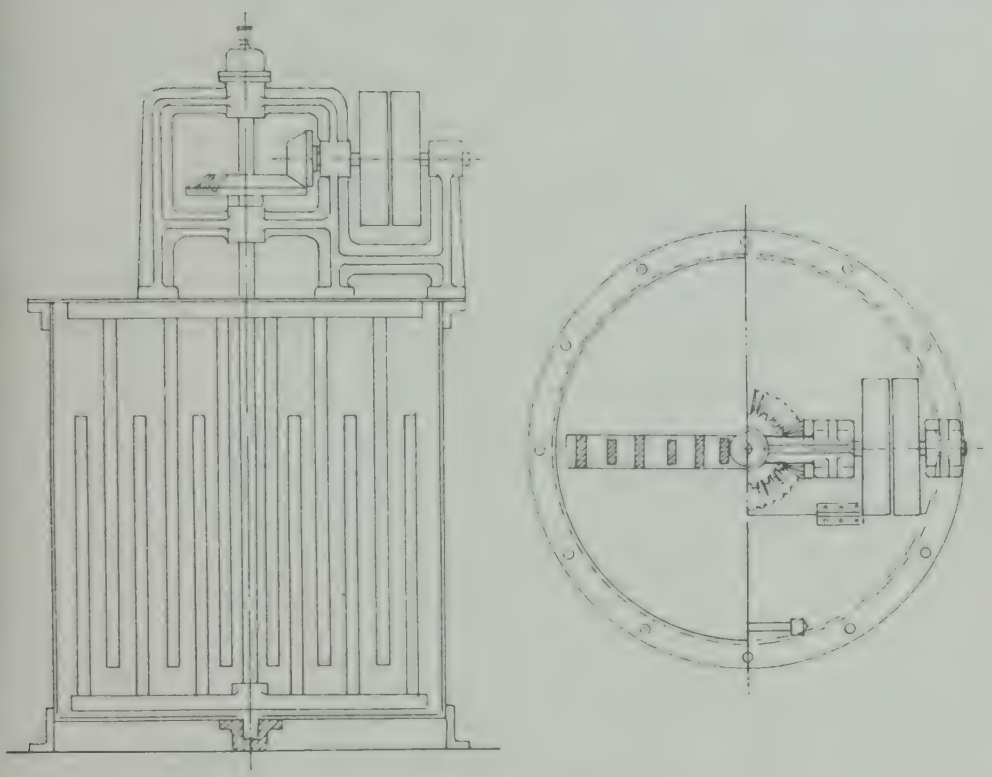


FIG. VIII–7.—HEAVY-DUTY MIXER.

to break up lumps is the "Muller" or pan mixer. This mixer acts as a combined crushing and mixing plant and finds very wide application. Two heavy rollers are driven from a central shaft and rotate on a circular track. A plough running in front of each roller scoops up the material, turns it over, and feeds it back under the following roll. These mills are used in such diverse operations as the mixing of chocolate aggregate and the compounding of stone-bitumen mixtures for road-surfacing. The design is very old—these were probably the first large grinding plants used by man—and it is possible that the wide application is due to tradition rather

than general utility. Any other batch grinding process, such as ball milling, will serve to mix solids together. When the solids have already been ground, a lighter construction than that required for ball milling can be used, with an irregular form so that cascading of dry solids is irregular. A stirring device may be incorporated to make sure that no sticking of the charge occurs, and a typical mixer of this design is shown in Fig. VIII–8. This is a rotating cylinder, turning in the opposite direction to the helical ribbon mixer inside. A cylinder 4 ft. in diam. by 7 ft. long will hold a charge of 29 cwt. of solid and take 10 h.p. for driving.

When agitation is to be improvised on existing vessels, compressed air is generally used. A distributing pipe at the bottom of the

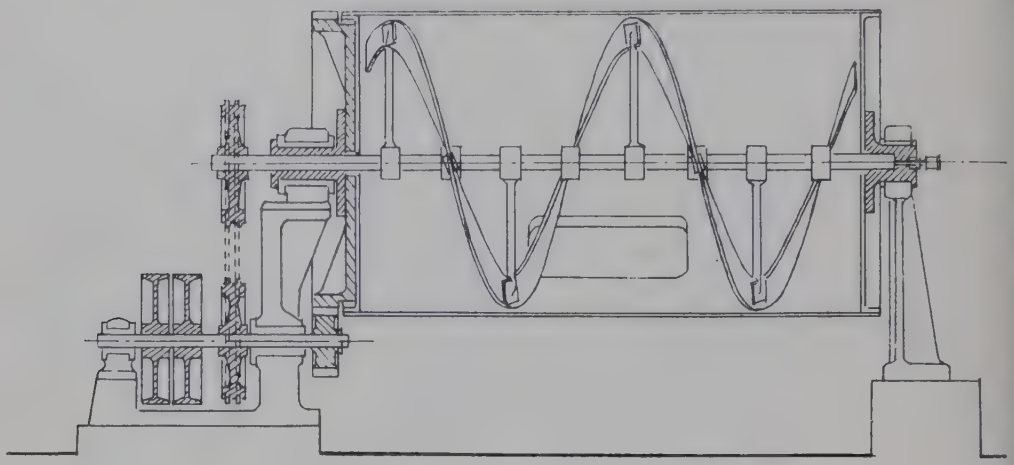


Fig. VIII–8.—Powder Mixer.

vessel spreads the air as thoroughly as possible, and a reasonable makeshift agitation, sufficient to prevent light solids from settling, is obtained. If air agitation is necessary for some reason, such as the avoidance of all moving metal parts in explosive manufacture, it is usual to arrange for some form of air lift to circulate liquid from top to bottom of the mixture. Where foam formation is required, a turbo-agitator with a hollow shaft will draw air down into the centre of the impeller and there discharge it along with the liquid. Smaller aeration units in which the air, or other gas, is reacting with the liquid, can be built up from porous porcelain tubes. These tubes, mentioned in the chapter on Air Filtration, are mounted on ebonite or steel formers, and gas is blown through under pressure. A suitable compromise has to be made between the amount of air passing unit area and the degree of dispersion required.

A pump method of mixing which is sometimes used consists in drawing off liquid from the base of a tank and returning it to the top. It is considered to be more costly in power than stirring, but can be combined with heating or cooling of the circulating liquid. The heat-transfer device can be outside the tank, and by suitable pipe connections can be made to serve a number of tanks intermittently. The pumping action may create a swirl in the tank and draw in two liquids simultaneously, and then "homogenise" the mixture by an adjustment of the pump outlet valve. Alternatively, another constituent may be drawn in through a venturi throat by the jet action of the pumped fluid.

CHAPTER IX

CRYSTALLISATION

The importance of crystallisation as a technical process has been partly obscured in literature by an enormous amount of work upon theoretical aspects of crystal structure. A simple crystallising process can isolate a pure compound from a mixture and at the same time produce this compound in the final form in which it may be handled or sold. Sugar, or ammonium sulphate, are typical cases of this form of processing. Only a pure product will give a satisfactory crystal, and it follows that a large well-formed crystal carries with it a certificate of reasonable purity.

Crystals grow from a liquid phase when limits of solubility are passed, provided that some nucleus for crystal growth is present. The problem of providing such nuclei varies enormously with the compound being crystallised. Many salts form nuclei and start crystal growth almost as soon as supersaturated conditions obtain, while others may need such a high degree of supersaturation that no crystallisation normally occurs, and the solution may solidify as a supercooled liquid or glass. This last state is very common with organic materials, though obviously, as the name implies, not unknown with inorganic salts. It is probable that all solutions exhibit the normal stages of Fig. IX–1, in which a metastable zone is located between unsaturated solution and solid–liquid region. In this zone nucleus formation may be very slow and crystal growth will be around such nuclei as are already present. When pure liquids are cooled to below their freezing-point, solid nuclei form and then grow more or less rapidly to large crystals. The rate of formation of these nuclei is found to follow a curve (Fig. IX–2) which, as the temperature is steadily lowered beyond the true melting-point, reaches a point of maximum rate and then falls off again. Substances cooled rapidly to well below " maximum rate temperatures " will solidify to a non-crystalline solid, as suggested above. It is probable that similar rates of nucleation could be observed with solutions if it were possible to obtain exact measurements, but

normally the control of nucleation in solutions can only be achieved by keeping conditions in the metastable region. The working of a controlled crystalliser is chiefly concerned with the growth of crystals on existing nuclei and with the prevention of fresh nuclei formation. Only in this way can a crop of large even crystals be obtained.

When a crystal nucleus is present in a supersaturated solution it will grow at a rate which is determined by three main factors—degree of supersaturation, rate of movement of liquid, and specific surface factor characteristic of the crystal substance itself. For

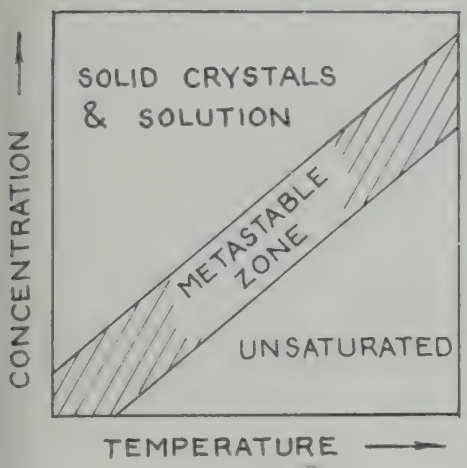


Fig. IX-1.

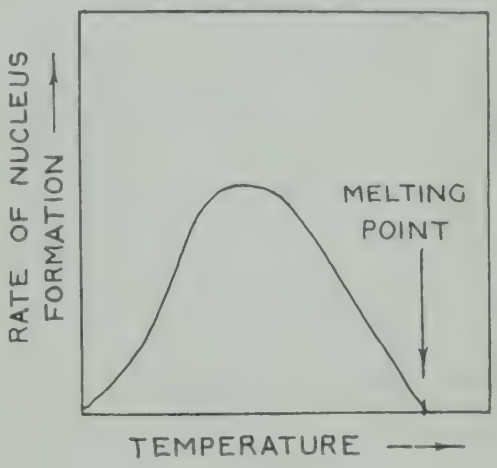


Fig. IX-2.

any one crystal the rate of deposition of solid, subject to these factors, can be assumed as proportional to the actual surface, or

$$\frac{dW}{dT} = K_s \times f(C - C_0) \times M \times A \quad . \quad . \quad . \quad (1)$$

where K_s is the surface constant, $C - C_0$ the degree of supersaturation, M a measure of the liquid movement or mixing, and A is the crystal surface area. The weight of any solid particle of density ρ and linear dimension L will be $K_1\rho L^3$, where K_1 is a constant. Similarly the surface A will be K_2L^2. Then $W = K_1L^3\rho$ and $dW = 3K_1L^2\rho dL$. Substituting these values in equation (1)

$$\frac{3K_1L^2\rho dL}{dT} = K_s \times f(C - C_0) \times M \times K_2L^2$$

or

$$\frac{dL}{dT} = \frac{K_sK_2}{K_1} \times \frac{f(C - C_0) \times M}{3\rho}$$

The expression on the right-hand side of this last equation is independent of L and it follows that the change in linear dimension of a growing crystal is not proportional to the size. Thus a set of growth conditions which cause a $\frac{1}{16}$-in. crystal to grow to one of $\frac{3}{16}$ in. will also cause a $\frac{1}{4}$-in. unit to grow to $\frac{3}{8}$ in. If this mathematical analysis is sound, then it should be possible to calculate the size of crystal to be produced from a given size range of seeding crystal by further removal of solvent or drop in temperature. In practice this is not the case, for a variety of reasons. In the first place the rate of deposition is not evenly proportional to all the crystal surface, and each facet must have a different rate of growth if crystal shape is to be preserved. Secondly, the process of crystallising always produces new centres of growth, either through breakage or spontaneous nucleation. Various other factors, such as the presence of colloidal impurities, combine with the two principal causes to make calculated-size figures very dubious.

Rates of crystal growth are determined finally by the degree of supersaturation, which in turn is controlled by rate of removal of (*a*) heat or (*b*) solvent. For controlled crystallisation these rates must be so regulated that local concentrations are kept within the metastable range of Fig. IX–1, or a fresh crop of nuclei will form. If crystal size is not important, then fresh nucleation does not matter and rates of crystal growth are only limited by heat transfer and similar problems. Any large evaporating plant will therefore serve as a crystalliser for small crystals, or in certain cases for irregularly shaped crystal masses. When large crystals—in practice anything of above 0·5 mm. in size—have to be grown, then rates must be more carefully controlled.

The usual plant for uncontrolled crystallisation is the " salting " evaporator. This is a normal calandria evaporator, preferably with short wide tubes and a large central downtake, with the base drawn out to a cone and fitted with either a salt box receiver or a barometric leg discharge—the latter being the more common. In the salt industry, which produces more fine crystals than any other branch of chemical technology, a copper- or monel-tubed evaporator produces a slurry containing up to 20% of solid NaCl crystals. This flows down through a barometric leg to the feed tank of a " top feed " rotary vacuum filter, and the filtrate is returned to the evaporator. The principal impurity in the sodium chloride used is calcium chloride, which accumulates in the evaporator until a high

concentration is reached. The whole evaporator contents are then discarded. This is a typical instance of purification by crystallisation. Ordinary evaporator operation will produce crystals of the order of 0·2 mm., with some variation according to the salt being separated. The crystal size can be increased if the crystal nuclei are circulated as efficiently as possible through all parts of the evaporator. Normally the small crystals settle rapidly into the coned bottom, but a paddle or propeller stirrer can force the slurry

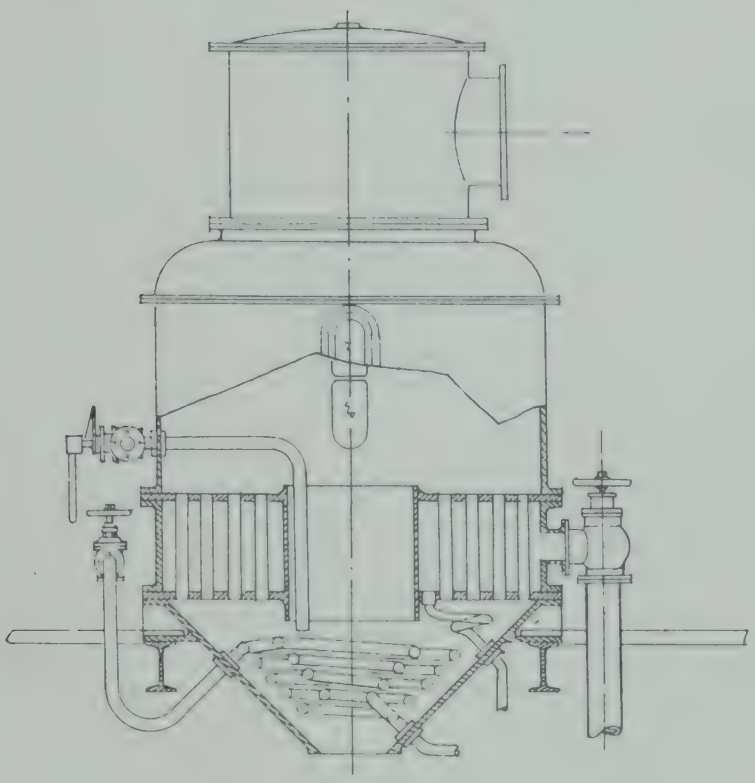


Fig. IX–3.—Vacuum Pan Crystalliser.

round as in forced circulation evaporators. Unfortunately the agitation tends to break up the crystals, but it is possible to increase the size of NaCl to some extent.

In the sugar industry, where a definite crystal size is more important, though a fine crystal is still required, a batch process is used. The sugar solution is evaporated almost to crystallising point in ordinary multiple-effect evaporators, and then fed to a finishing pan, or " vacuum pan ". This (Fig. IX 3) is an evaporator heated either by coils or by a short tube calandria with a large central downtake –up to 50% of the area of the boiling tubes as against

20% for an ordinary evaporator. A charge of solution is concentrated until nucleation begins, and then "struck", by increasing vacuum and cooling by flash evaporation, until sufficient nuclei are present for all subsequent crystal growth. This process is controlled by the experience of the skilled plant operator in charge, and has been based on trial and error over a number of years. When nucleation is complete, fresh sugar solution is fed in and evaporation continued until the pan is filled by a thick suspension of sugar and solution. This charge is then dropped either to further cooler

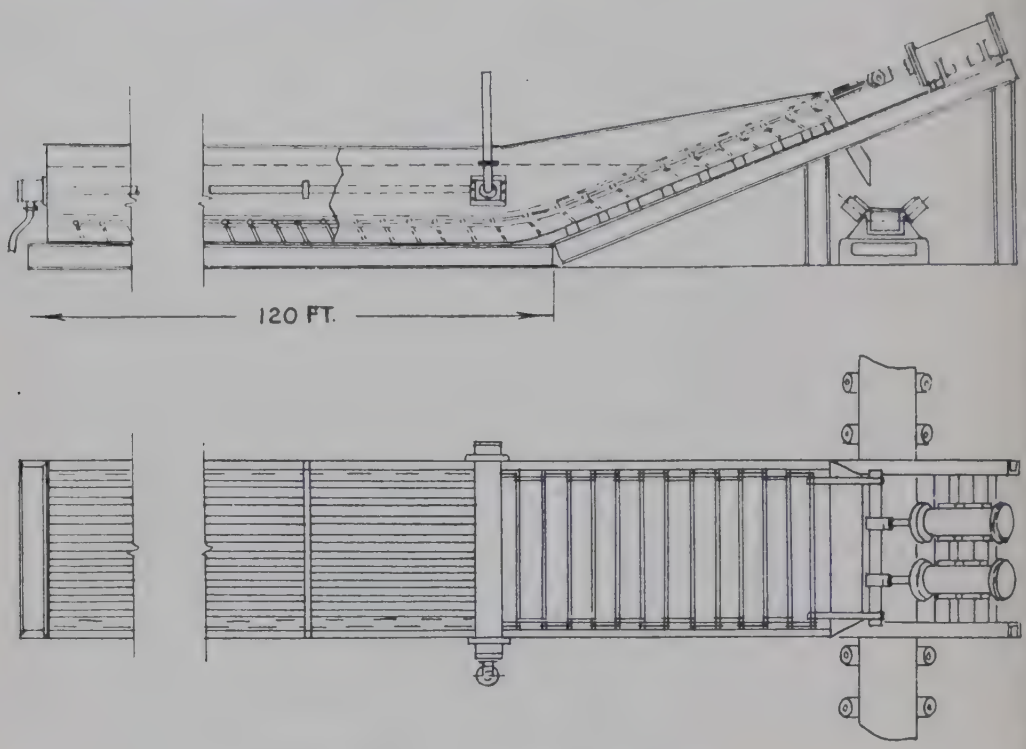


Fig. IX–4.—Fishery Salt Pan.

crystallisers or direct to centrifuges. Sugar-boiling to a definite crystal size is still an art rather than a science, but the varying amount of other organic impurities present will always make rigid control difficult. However, most sugar crystals are grown to a relatively small size—about 1 mm.—and the larger sizes are grown in the stirred crystallisers referred to later.

The fishing industries of the world have for hundreds of years used sodium chloride for fish-preserving, and this "fishery salt" has a special crystalline form. It is ideally a saucer-shaped crystal of 5–10 mm. across, and is produced by slow evaporation of the salt solution in large open pans of the form shown in Fig. IX–4. These

long shallow tanks are heated by steam coils, or even by direct firing with hot gases passing along heating flues under the tanks. The liquid is held at just on boiling-point, and convective air currents carry off water at a rate of 5–10 lb. sq. ft. of liquid surface per hour. The solubility of NaCl is almost independent of temperature, and crystallisation is therefore initiated at the liquid surface as the solvent is removed. The crystal nuclei are held at the surface by liquid tension effects, and grow at the edges, forming the characteristic saucer shape. The size increases until the weight is sufficient to break the surface skin, when the "hopper" falls to the tank bottom and is removed by rakes. The crystals are hard, difficult to dissolve, and have a low packing density. The operation is far from ideal, being costly in both heat and labour, but no alternative process has yet been devised to grow sodium chloride crystals in this or any other forms possessing the properties detailed above. This is probably due to the marked ease with which the salt forms fresh nuclei with a very small degree of supersaturation, and to the very small change of solubility with temperature.

When the solubility characteristics of salt are suitable, a number of relatively simple cooled crystallisers have been utilised. The cheapest and simplest method of all is to run a hot saturated solution into open pans and leave to cool by atmospheric action. As rates of cooling are then slow, especially with large vessels, crystals of considerable size can be grown, though they tend to be irregular in form, and often interlock to a solid mass which must be broken up by hand before it can be removed from the crystallisers. For ease in this the crystalliser can be built as a shallow trough with a sloping end, from which crystals can be raked during the cooling process, as with fishery salt, or dug out by spades when cooling is complete. In a typical case, that of aluminium sulphate, the hot concentrated solution is run from the reactor-concentrator into long troughs of acid-resisting brickwork, 6 in. deep and 2 ft. in width, and left to solidify. The interlocking crystal mass is deeply scored into 12-in. squares at an intermediate pasty stage, and can be broken up with ease when fully set.

Clearly these crystal growths are very irregular, and for properly formed crystals some type of stirring is necessary. To increase rates of production it is also necessary to water-cool the solution. Stirred cooled crystallisers have been built with many different designs—"as numerous as the engineers who have been concerned

in their development ".* The principal difficulty is to keep the cooling surfaces free from solid growths, and the most popular designs provide for scraping stirrers. Such scraping action is almost bound to break up the small crystals, and thus increase the number of nuclei and the possible sources of distorted crystals. The problem of crystal adhesion to a surface has not been very fully studied; there is no positive reason why the solid should adhere, and probably all sticking commences as a local corrosion effect. If the cooling surface is perfectly inert to the solution, then no crystals should adhere. So far no very inert surface has been put into use in crystallisers, but polished stainless steel, kept in condition by periodic

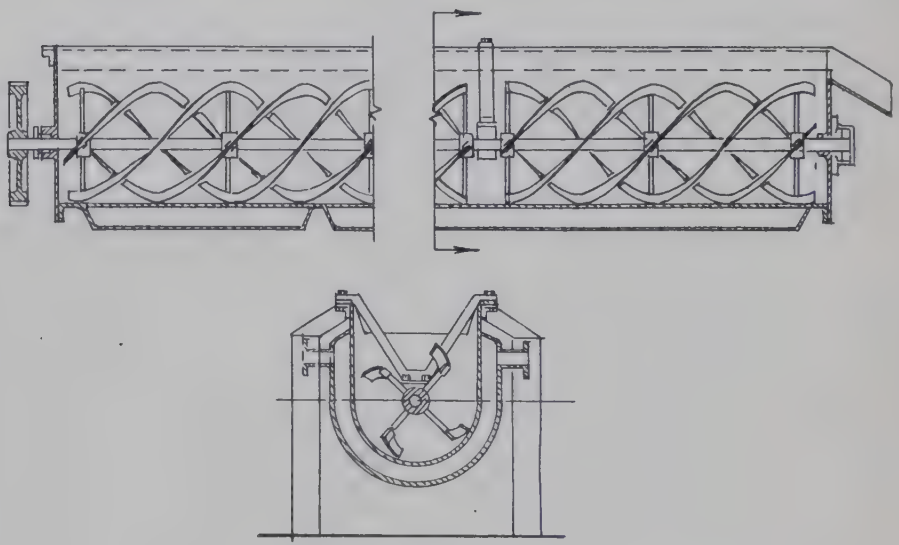


FIG. IX–5.—SWENSON-WALKER CRYSTALLISER.

scouring and burnishing, will be fairly satisfactory. The ideal material would perhaps be the new plastic "Teflon",† which is claimed to be inert to all chemicals with the exception of molten sodium, while rubber covering might also be applied.

Probably the most widely used crystalliser of the stirred type is the Swenson-Walker, as shown in Fig. IX–5. It consists of a U-shaped trough, 24 in. wide and up to 15 ft. long, with a water jacket divided into sections so that differential cooling may be used in the various zones. A close-fitting spiral stirrer keeps the crystal suspension in motion, and the solution is fed in at one end and overflows at the other. Nucleation may be started by a short

* Seavoy and Caldwell, *Ind. Eng. Chem.*, 1940, 32, 727.
† Renfrew and Lewis, *Ind. Eng. Chem.*, 1946, 38, 870.

cold zone, followed by graduated cooling, slow at first because of the relatively small amount of crystal surface present. Two or more of these long tanks may be arranged in series, and the discharged slurry can be fed to a centrifuge or top-feed rotary filter. Output will clearly be determined by the solution being handled, but is about 6 tons per 24 hours for a triple-tank system 30 ft. in length, while growing crystals of up to 0·25 in. in size. It is possible to cool solutions to well below atmospheric temperature by the use of

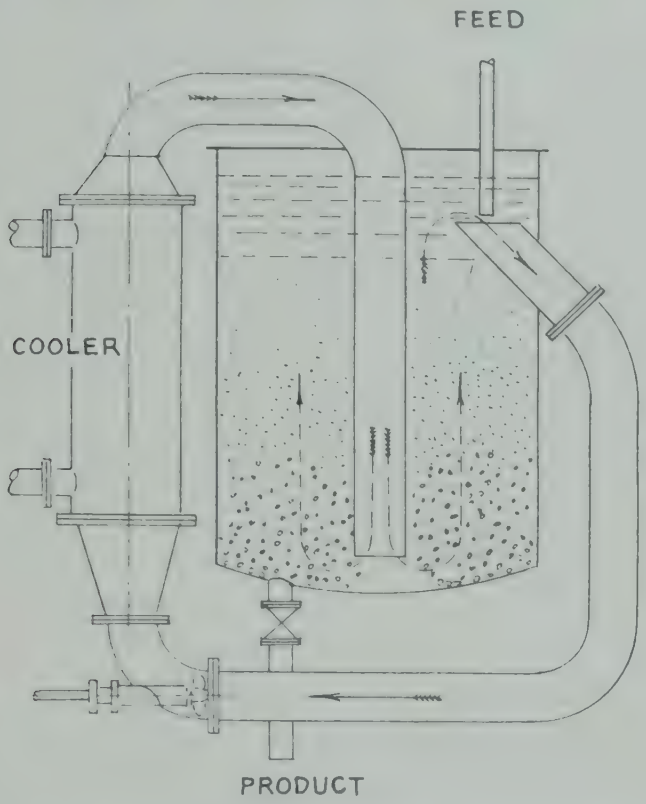


Fig. IX-6.—Forced-Circulation Crystalliser.

refrigerated brine, or even by the direct expansion of refrigerants in the cooling jackets. Obviously the only salts which can be crystallised in this way are those which have a marked variation of solubility with temperature, but a wide range of crystallising temperature can be obtained. This is an advantage over both atmospheric cooling and, as will be seen, vacuum crystallising.

It is possible to avoid stirring by a rapid circulation of the solution itself by means of pumps. The device is much the same as is used in forced-circulation evaporators, the liquid being pumped so fast through the cooling system, as shown in Fig. IX-6, that the crystal

growth does not occur until the supercooled liquid has reached the static crystal bed in the body of the crystalliser. This type of plant has been more developed in connection with vacuum crystallisation, and will be dealt with more fully below.

Another alternative to the Swenson-Walker is the Wulff-Bock rocking crystalliser. This, as shown in Fig. IX 7, has a trough of elliptic form, mounted on circular bearings, which allow the trough to be rocked to and fro through a total angle of about 90°. This rocking keeps the crystals in suspension and the fluid in motion without the need for a stirrer. Cooling is atmospheric, which limits temperature ranges and tends to give smaller throughputs than water-cooled units. The saturated solution enters at one end

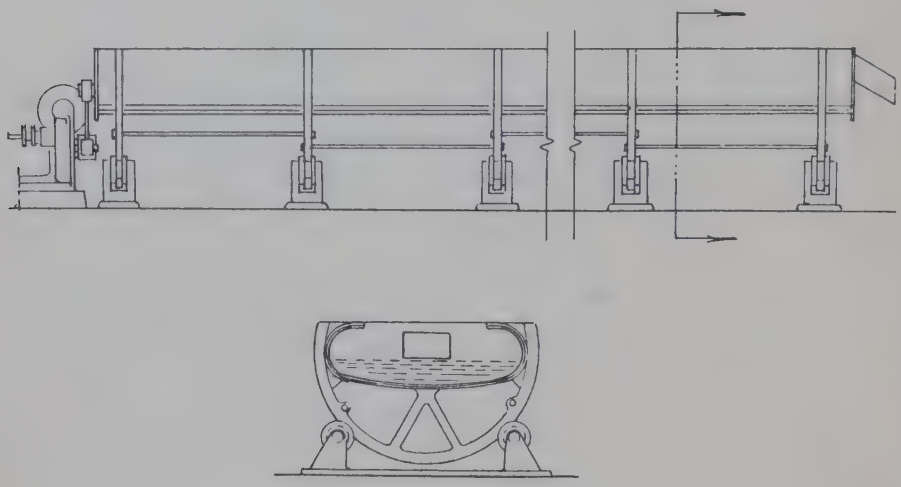


Fig. IX–7.—Rocking Crystalliser.

of the trough, which may be as much as 50 ft. in length, and flows through a circuitous path arranged by baffles to the discharge end. The absence of moving metal parts inside the working space makes the unit easier to construct in rubber-covered steel or similar anti-sticking materials, and the slower cooling with avoidance of scraping is claimed to give larger and more uniform crystals. It will be appreciated that with this and other forms of crystalliser the output in unit time falls with increase in the size of crystal required. An output of 2–3 tons per 24 hours can be obtained with such salts as sodium sulphite, using a rocking trough 3 ft. wide and 50 ft. long. The crystal size range will be from 0·25 to 0·5 in. In any particular problem the degree of supersaturation must be matched with the amount of crystal surface available and the efficiency of agitation required to move the crystals through the solution.

When the crystals required have a negligible temperature coefficient of solubility, cooled crystallisers are useless, and it is necessary to remove the solvent by evaporation. This means heating to boiling-point, or near it, and the low temperatures which are often necessary to grow hydrated crystals can only be secured by vacuum operation. This introduces the difficulty of condensation of low-pressure water vapour; if normal water-cooled surface condensers are used, the minimum boiling temperature in the crystallising

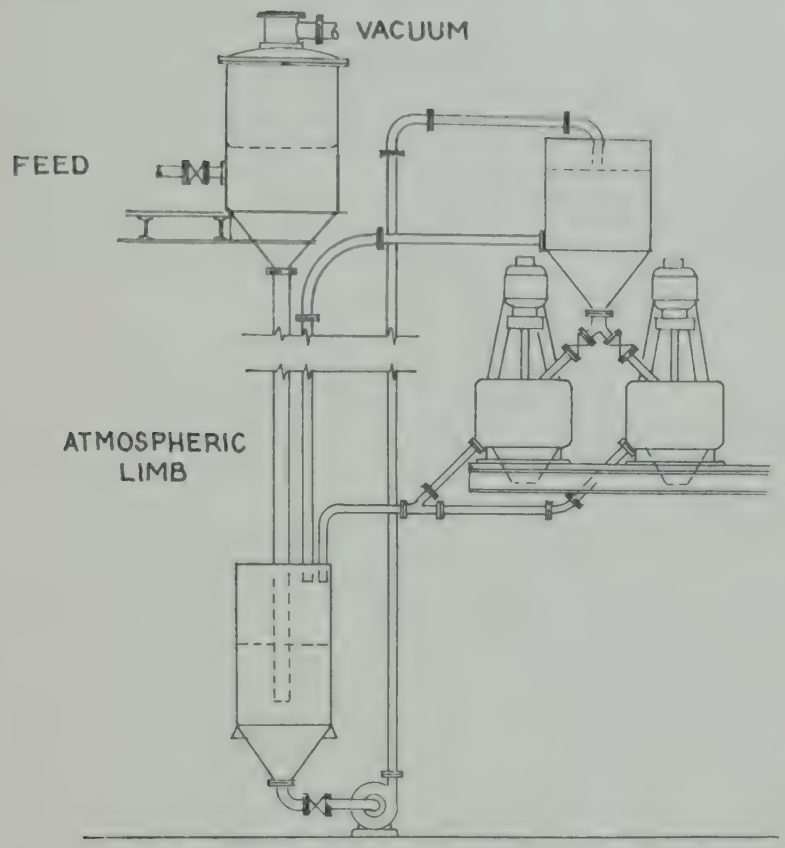


Fig. IX–8.—Vacuum Crystalliser.

process will be about 40° C. The only way in which lower temperatures can be conveniently attained is to compress the water or solvent vapour before condensation, a device which increases the complexity of the plant. Jet condensers present the same difficulties, but can be used in part to provide the compression necessary. Special refrigerated condensers could be used, as in freeze-drying, but their use has not been reported so far.

Despite this limitation on temperature, the use of vacuum crystallisation now appears to be spreading rapidly. Of course, the

vacuum pan of the sugar industry, referred to above, is a type of batch vacuum crystalliser, but the more popular form is one in which continuous feed of saturated solution and of withdrawal of crystal slurry makes for a regular output. In early forms of this plant, as shown in Fig. IX–8, a hot saturated feed was flashed to vapour and a crystal slurry flowed down through the barometric leg to discharge

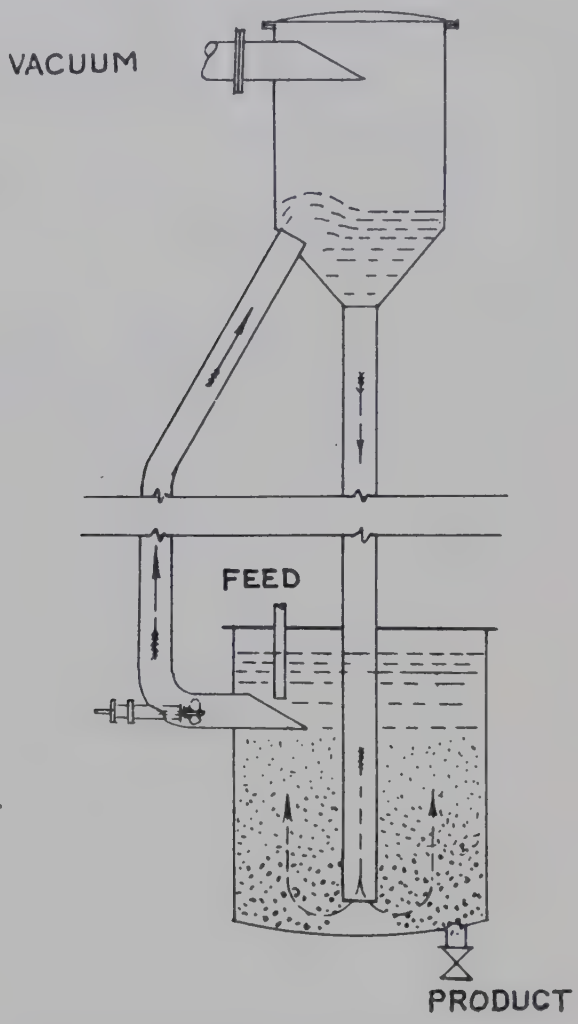


Fig. IX–9.—Forced-Circulation Vacuum Crystalliser.

and centrifuging. This demanded a nice balance of feed and product, while nuclei could be removed from the flashing zone before they had grown to a satisfactory size. This form of plant has therefore been replaced by " A. S. Krystal " units, in which the solution is circulated rapidly through a bed of growing crystals, over heating surfaces, and then through a flash chamber in which a small quantity of solvent is removed. As will be seen from Fig. IX–9, the slightly

supersaturated solution flows up through the crystal bed, completing the circuit and depositing the surplus salt. Fresh feed in the form of saturated solution is added at any suitable point. The process follows the rules of forced circulation evaporation, in that all boiling takes place after the solution has left the heating surfaces, and the amount evaporated per "pass" is a small fraction of the whole fluid stream—less than 0·1% in some cases.

It will be seen that the supersaturated liquid, after evaporative cooling, passes down a central tube in which crystal growth can occur, and it is necessary to arrange for high liquid speeds over a highly polished surface if crystal growth is to be avoided. When the liquid leaves this tube it passes up through the bed of crystals at linear velocities of 2-5 ft./min., a sufficient rate to keep all the crystals in suspension and agitation. The crystal mass will probably "fluidise" or form an even suspension without size classification at these velocities, and in fact it has been shown that in one case* there is no even gradation of solid size. The same investigation suggested that for ammonium nitrate the degree of supersaturation of the liquid entering the crystalliser from the flash head should not be more than 0·01 lb./gal. This, for the amount of solution being circulated, corresponded to an evaporation of 0·003 lb. of water per gallon of solution entering the flash head. It was also necessary to direct the entering solution to give a swirl in the crystal bed, and to by-pass a small quantity to the upper levels, as shown in Fig. IX–10. In these circumstances a steady output of 50 lb./hr. of 8–12-mesh crystals could be obtained from a crystalliser of 150 gal. capacity, holding some 450 lb. of solid in suspension. The ratio of weight taken off per hour to weight in suspension appears to be critical. Such a design of plant offers every opportunity for exact control of supersaturation and crystal growth, and it is possible to base a design on first principles, though in view of the very individual characteristics of any specific crystal it will be desirable to check the figures experimentally.

Example. *A salt is being crystallised at* 45°-50° *C., at which temperature the saturated solution has a vapour pressure of* 60 *mm. Hg. The maximum supersaturation for avoidance of nucleus formation is* 0·02 *lb./gal. Suggest a design for a vacuum crystallisation plant to produce* 200 *lb. hr. of well-formed* 10-*mesh crystals.*

* Miller and Saeman, *Chem. Eng. Prog.*, 1947, 43, 667.

It may be assumed—with due mental reservation—that the ratio of weight of "make" to weight in suspension is the same as for NH_4NO_3. It follows that the crystalliser must have 2000 lb. of crystals in suspension, or about 650 gal. of solution. The amount of liquid which must be circulated to give 200 lb. of solid/hr. will be 200/0·02 or 10,000 gal./hr. This is 27·2 cu. ft./min., and assuming an upward current of 3 ft./min. in the crystalliser the cross-section must be 9 sq. ft. The height of a tank of 650 gal. capacity will therefore be 11·5 ft.; to this must be added

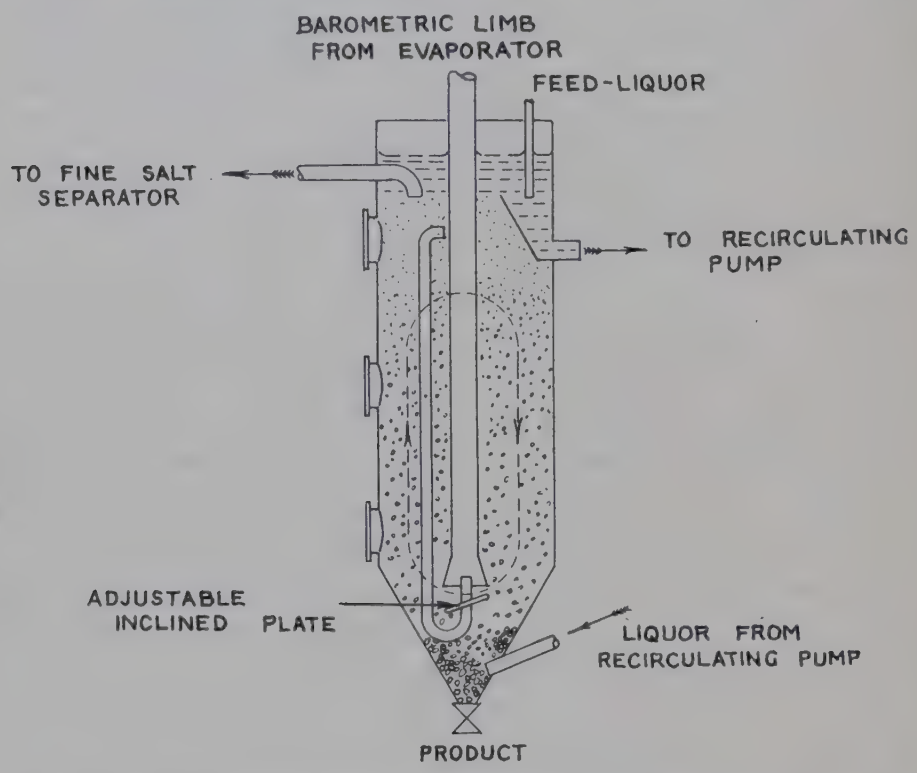


FIG. IX–10.—CIRCULATION IN AMMONIUM NITRATE CRYSTALLISER.
(Miller and Saeman, *Chem. Eng. Progr.*, 1947, 43, 667.)

a "clear zone" for the crystals to settle (Fig. IX–9) of perhaps 1·5 ft., giving an overall height of 13 ft.

The amount of water to be evaporated is 300 lb./hr., or 0·03 lb./gal. of liquid circulating. This would involve heating-up the liquid from the crystalliser by about 3° F. before discharging to the flash head. Alternatively, vacuum might be dispensed with and a current of air blown into the head to vaporise this very small amount of water.

Obviously a small crystalliser would have to be built and the assumptions made in the above example tested out before proceeding to the full-scale design.

A very important form of reactor-crystalliser is used in the commercial manufacture of certain salts, notably ammonium sulphate. In this plant or "saturator", ammonia gas from stills is blown directly into a saturated solution of the sulphate, and concentrated (70^{o}_{o}) sulphuric acid is run in simultaneously to maintain a free acidity of $1–4^{o}_{o}$ H_2SO_4. The salt crystallises out, is lifted on to drainage tables by a steam-jet lift, and then raked into centrifugals.

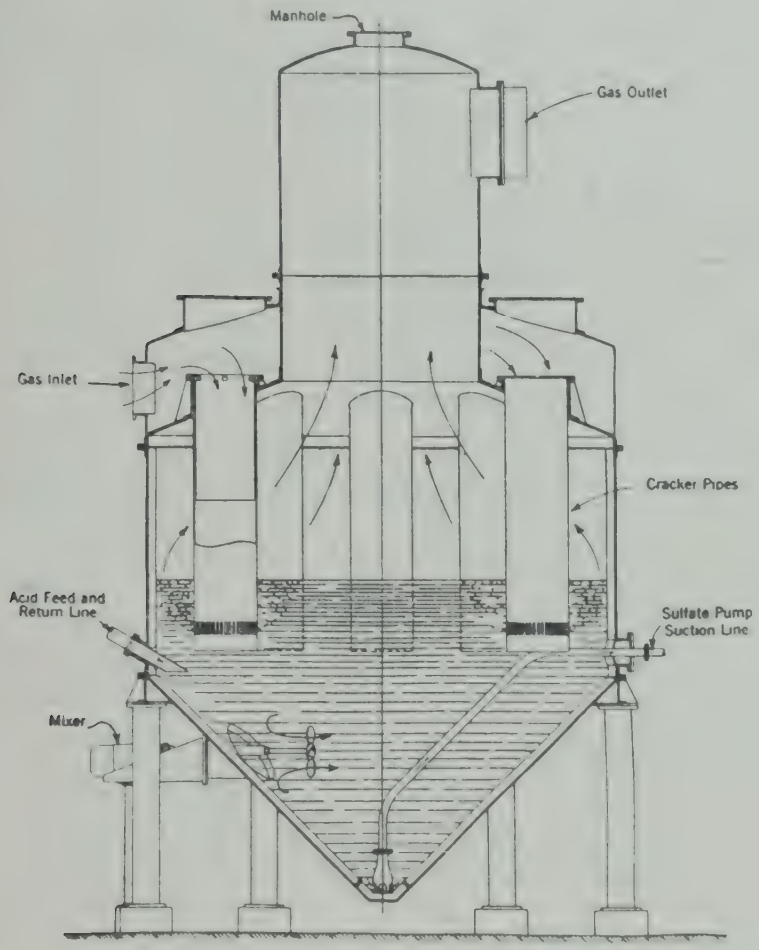


Fig. IX–11.—Ammonium Sulphate Crystalliser.

The layout of a typical reactor is shown in Fig. IX–11. The bulk of the plant is in lead, with the centrifugal basket in monel metal. A very similar type of plant, constructed in stainless steel, has been used in the manufacture of ammonium nitrate. These reactor crystallisers are just as sensitive to conditions as other types of crystalliser. The acidity must be held at a steady value, as alterations vary the salt solubility and tend to produce large crops of tiny crystals.

Another phenomenon which is often met with in crystallisation arises in connection with ammonium sulphate. This is the influence of impurities, both organic (traces of tar) and inorganic (other metallic salts). The ideal form of crystal in this case would be a cube of about 1–2 mm. side; larger crystals would not feed easily through the " drills " of fertiliser spreaders, and smaller sizes would be apt to cake together on storage. It has been found that pure $(NH_4)_2SO_4$ resembles common salt in that large crystals are not easy to grow, but the crystal form is almost cubic. When trivalent metal ions are present in the solution, the crystals can be grown to larger sizes, but lengthen to a needle form, which is easily broken and chokes machine " drills ". By the use of controlled quantities of the trivalent ions ($Fe^{\cdots}$ and $Al^{\cdots}$) it is possible to achieve a condition in which the ammonium sulphate crystal grows to a convenient size but is still in the form of short thick rods. Many other substances act to modify crystal growth. Gelatine in solution in water checks the growth of ice crystals, and it is common knowledge that impure organic substances will not crystallise, even though nuclei of the pure substance are provided. A paper* dealing with the effect of impurities on the crystallisation of sucrose showed that the rate might be cut from 200 gm./sq. m./hr. to less than 1 gm. by relatively high concentrations of organic colloids, but might actually be accelerated by small quantities of inorganic salts and invert sugars. This makes it absolutely necessary that figures for plant design must be based on experiment with the actual salt to be used.

* A. Van Hook, *Ind. Eng. Chem.*, 1948, 40, 85.

CHAPTER X

LIQUID FILTRATION

The separation of two phases—solid or liquid from liquid or gas—can be defined as a single-unit operation of filtration, although this term has usually been applied only to the separation of a solid from a liquid. The other separations may use the same methods, especially in the field of centrifugal practice, and the subject could therefore be treated as a whole, but for convenience has been divided into three chapters, Liquid Filtration, Centrifugal Practice, and Gas Cleaning.

The liquid filtration process separates solid particles from a suspension in liquid (a slurry) by forcing the liquid through a filter medium in which the pores are too small for the solids to pass. The pressure may be simply that of gravity, but is more usually provided by evacuating the space below the filter medium, or increasing the pressure above it by any suitable means. In the initial stages of the filtration the characteristics of the filter medium are of some importance, but the properties of the actual filter-cake, as it forms, soon begin to exercise a decisive influence. In almost all commercial filtration problems the rate of filtration and hence the size of the plant are almost entirely determined by the properties of the filter-cake. This filter-cake is built up from individual solid particles, which vary widely in all characteristics according to the composition of the solid and the way in which precipitation has been carried out. Even the simplest chemical precipitation can be varied to produce any solid form from sand-like grains to a jelly, with no general rules governing the form taken. Thus barium sulphate can be thrown down in coarse hard grains by hot precipitation from dilute solutions, or as a stiff jelly by mixing cold concentrated solutions of barium chloride and sodium sulphate. It is possible to modify the character of a solid suspension very markedly by the addition of colloids or the adjustment of acidity, and one of the difficulties encountered in the investigation of filtration has been that of obtaining a standard precipitate. An illustration of

this which came to the author's notice some years ago was the precipitation of naphthol from an alkaline solution after dissolving a fusion of caustic soda and naphthalene sulphonate. If the fusion was interrupted before completion, the naphthol, "struck out" by acid addition at about 100° C., was thrown down as a slime. As the sulphonate in the melt was reduced by continued reaction to below about 2%, the naphthol precipitated in solid droplets of up to 1 mm. in size. The difference in filtering time was from 2 days to 1 hour, and clearly plant designed for one slurry would be quite unsuitable for the other. As a result of such facts it cannot be too strongly stressed that the general principles of filtration cannot be applied to particular cases without a great deal of experimental data dealing directly with the slurry being considered.

The flow of a liquid through the narrow channels of a filter-cake and filter medium is clearly analogous to flow through a capillary tube. The rate of flow will then be proportional to the applied pressure and inversely proportional to tube length (thickness of cake) and fluid viscosity. As filtration continues, the thickness of the deposited cake increases, increasing the length of the equivalent capillary tube. This hypothetical length can be given a resistance to flow R such that, for a fluid of fixed viscosity, the instantaneous rate of flow dQ/dT for unit area of filter is given by $dQ/dT = P/R$ (where P is the applied pressure). The total resistance R will be built up from the filter medium resistance A and a variable resistance due to the filter-cake, proportional to the amount of fluid Q which has passed, and denoted by BQ. Then $dQ/dT = P/(A + BQ)$ (where A and B are appropriate constants). This equation may be integrated between the limits O, T and O, Q to give the general expression for filtering rate

$$PT = AQ \times BQ^2 \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

This is an equation for the rate of filtration of an ideal slurry under constant pressure. By ideal slurry is understood one which does not alter filtration characteristics with varying pressures owing to deformation of solid particles in the filter-cake. The constants A and B require experimental determination and can be related to any one of the various functions of Q. These are cake thickness or weight, filtrate volume, slurry volume, and so on. With a slurry of constant composition there will be a fixed relation between all such quantities.

There is, for any solid material in a liquid suspension, a limiting pressure beyond which further increase, by deformation of the solid cake, may not proportionately increase filtration rate, and may in certain cases decrease it. This "critical pressure" may be very high and well above normal filtering pressures, but it can be so low that only gravity filtration can be used. Again, if the solid particles vary in size, then the flow of fluid through the gaps between the larger fragments carries the finer material up to the immediate neighbourhood of the filter medium to form a subsidiary layer of low porosity. This will increase the resistance to flow over the value based on simple quantity measurements. It has been suggested, therefore, that the simple equation (1) be modified to the form

$$P^{1-s}T = AQ + BQ^m \quad . \quad . \quad . \quad . \quad . \quad (2)$$

where s and m are further values to be experimentally determined. For most industrial slurries it has been shown that s is small, while m approximates closely to 2.* A further simplification can sometimes be made when the resistance of the filter medium is negligible compared with that of the filter-cake. In this case (1) reduces to

$$PT = BQ^2 \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

This brief statement of the fundamental theory of filtration has assumed that all filtration is carried out at constant pressure, whereas the earlier stages of many filtrations are at constant rate. Fuller developments, covering this and other abnormalities, can be found in more detailed works on the subject.

The type of filtering plant to be chosen will depend upon the nature of the slurry, the amount of material to be handled, and the relative costs of labour and of plant. The following are the main types of filter available:

Filter-Presses. Ideal for slurries containing less than 5% of solids. Clumsy for large quantities. High labour charges.

Pressure-Leaf Filters. Same slurries as filter-presses. Lower labour charges, but still not adapted for large quantities.

Rotary Vacuum Filters. Ideal for slurries containing 15–30% of solids. Used for all large filtering operations if possible. Low labour charges.

* See, e.g., Sperry, *Ind. Eng. Chem.*, 1944, 36, 323.

Edge Filters. Ideal for clarification problems, with liquids containing small quantities of finely divided solids.

Other types of filter are sometimes met with, but are relatively unimportant.

Filter-Presses. These are the most commonly used of all the filter plants above. They consist of a large number (up to 60) of filtering chambers arranged in parallel on a suitable framework. In Fig. X–1 a recessed-plate filter is shown in section, while in Fig. X–2 the same filter is shown assembled. It will be seen that each pair of plates forms a recess lined on each side with filter-cloth; into this recess the slurry is pumped through the central channel A–A–A. As the pressure of pumping develops the filtrate

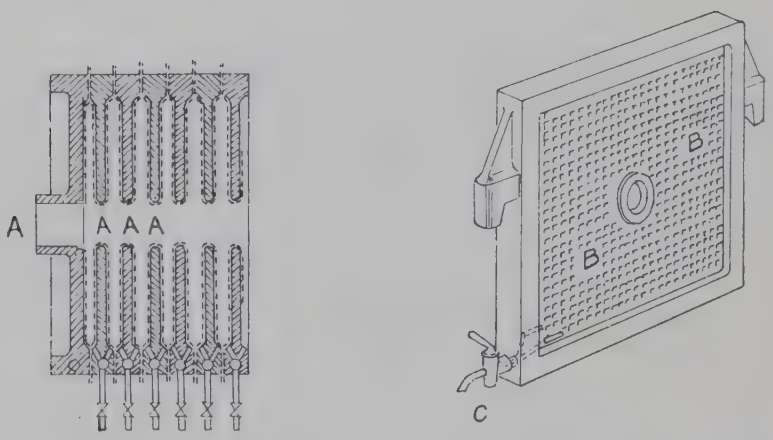


Fig. X–1.—Recessed-Plate Filter-Press.

is forced through the cloth into the channels of the plates B, B, B, leaving at the outlet taps, C, C, C. The solid builds up at each side to form the filter-cake, and finally the two cakes meet in the middle to fill the whole of the recess. This is a cake of predetermined thickness, according to the design of the press, and will be varied according to the characteristics of the slurry being filtered. In general, the easier the filtration the thicker the cake, and the plates will be constructed with deeper recesses for sand-like precipitates than for fine slimy deposits. The thickness varies from 0·5 in. to 3 in. but is not usually more than 1·5 in., and another type of press —the plate-and-frame—is used for the thicker cakes. It is not usual to arrange for cake washing in recessed-plate filters, and they are generally designed for simple filtration with slurries of indifferent filtering qualities. The design lends itself to high pressures rather

better than the plate-and-frame type described below, although the joints are more difficult to make fluid-tight. The form of the filtrate channels is so chosen as to give good support to the cloth and at the same time to give free flow to the filtrate. Various forms are used, according to the material of the plate and the techniques of the manufacturer. The most typical constructional material for these plates is cast iron, but any other machineable metal can be used, and wood plates are common. As there are no moving metal parts in actual contact with the filtrate or slurry, corrosion problems are

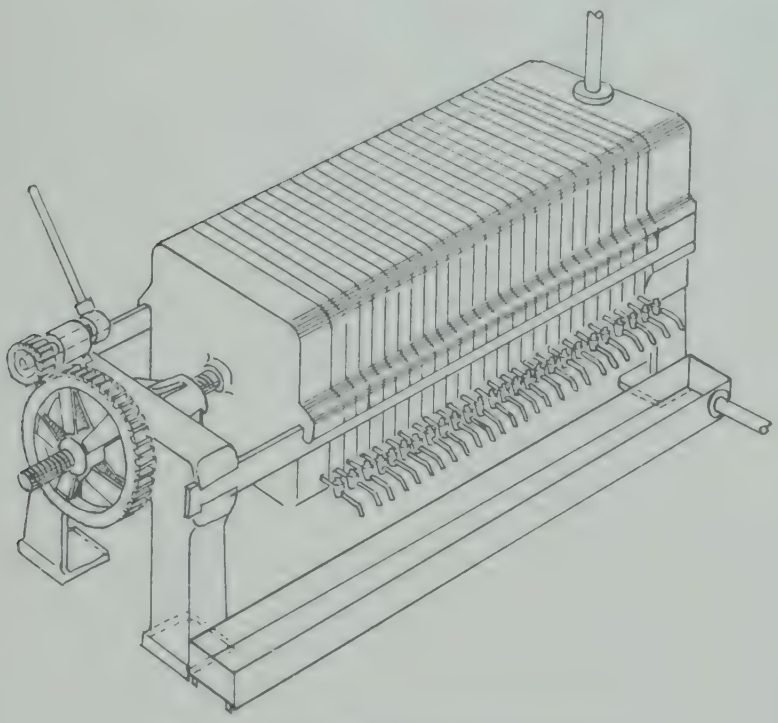


FIG. X–2.—ASSEMBLED FILTER-PRESS.

simplified, and the plates may be surfaced with a corrosion-resisting coating.

The weakest point of the press is the filter-cloth, which has to serve the dual purpose of jointing to a fluid-tight system and of forming the initial filter medium. The shape of a recessed-plate filter makes for some distortion of the cloth. The cloth is pressed hard against the corrugations forming the filtrate channels, and then irregularly distorted at the rim of the plate, being finally jammed between two metal surfaces. The normal method of making up these filter-cloths is to join two cloth squares in the centre, round the rim of a hole to fit the slurry channel. The joint is best made by

some special circular clip, as sewing puts an extra strain on the cloth. The distortion of the cloth fibres at the plate rim is considerable, and this increases with cake thickness; it is for this reason, among others, that recessed plates are normally designed for thin cakes. Conditions in plate-and-frame units are a little better, but the cloths still fail at the "joint" sections. Nevertheless, it has been found more economical to use the cloths for jointing rather than build in special jointing rings of rubber, with the filter cloth mounted as an interior mat.

If the rate of discharge of filtrate in a press operation is plotted against time, a curve of the type shown in Fig. X–3 is produced. A short initial period of irregular rate is followed by a steady "falling rate" section, with conditions governed by equations of the type developed above. This period terminates more or less abruptly as the two sides of the filtering space touch, and is followed by cake compacting, with a sharp diminution in discharge rate. In the initial period such factors as air discharge and the irregular swirl with which the slurry enters the empty compartments play a great part in determining filtration rate. It is theoretically possible to calculate values of A and B in the equation $PT = AQ + BQ^2$ from two values of T and Q, such as the times taken to form a 1-in. and a 2-in. cake. These values can then be used to calculate the time required for any given cake thickness. This is not to be recommended, as only the middle section of the filtering period is governed by a specific equation. The most satisfactory method is to extend experiments over the entire range of cake thickness for which data are required, and to use rational extrapolation methods based on a graph of filtering time against cake thickness. Values calculated from an equation can be used to give an indication of filtration time and its effects upon design, as will be seen later.

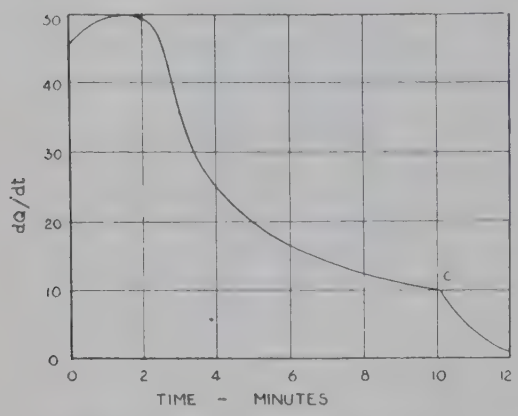


FIG. X–3.—FILTERING RATES.

When the filtering period is complete, as at some point C on Fig. X–3, the pumping system is shut off and the pressure discharged —usually by means of a by-pass on the pump inlet. The press is

now opened by screwing back the foot-plate (Fig. X.-2), the first cake is exposed and often drops directly from the cloth into a tray or truck below. If need be, it is scraped from the cloth by wooden scrapers, the amount of work required varying with the nature of the cake. The next plate is then pushed along the side rails to the foot-plate, exposing the second cake, and so on until the whole press is emptied. Provided that the cloths do not now require washing or other attention, the press is reassembled, drawing up the plates in reverse order. Each plate is adjusted with the filter-cloth joint spread smoothly between the rims—a process requiring considerable care—and finally the foot-plate is screwed back to lock the whole plate bundle into a fluid-tight unit. All this is very laborious, expensive in time and labour, and far from clean, even with skilled workmen. Clearly the press should handle as much slurry as possible between each opening, and this is the reason for the specification of dilute slurries made above.

It is usual to design a filter-press to contain a given weight of solid. The number of chambers required will then be inversely proportional to the cake thickness chosen, as 10 cakes of 4 in. thickness will weigh as much as 10 cakes 1 in. thick. Now the emptying cycle of any press will be dependent upon the number of chambers to be cleared, as well as on the nature of the cake. Consider a filter-press of N chambers, each of thickness Q, which is to hold a given weight of solid. Then the total thickness D, or NQ, will be constant, whatever the value of Q chosen. If it is specified that the press be worked under a constant pressure, then the constants A and B may be modified to include the pressure, and the filtering time for any thickness Q will be given by $T = AQ + BQ^2$. If a time E is taken to discharge each cake, then the emptying time is NE, or the total cycle time is $AQ + BQ^2 + NE$. This supposes that the value for NE includes the time for reclosing the press. Now the total output will be NQ and the output in unit time will be

$$\frac{NQ}{AQ + BQ^2 + NE} \text{ or } \frac{D}{AQ + BQ^2 + DE/Q} = S$$

This output rate will be a maximum with respect to Q when $dS/dQ = 0$. That is

$$-D(A + 2BQ - DE/Q^2) = 0, \text{ or } AQ^2 + 2BQ^3 - DE = 0$$

For small values of A a simple solution to this cubic equation is given by

$$Q = \sqrt[3]{\left(\frac{DE}{2B}\right)} - \frac{A}{6B} \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

It may also be noted that where A is quite negligible $DE = 2BQ^3$. Substituting for D the equivalent NQ, $NE = 2BQ^2$. Now NE is the emptying time and BQ^2 approximates to the filling time. On this basis the emptying time should be twice the filling time for maximum rate of production of solid.

Example 1. *A filter-press is required to filter batches of $CaCO_3$ slurry containing 1 ton of dry solid. Tests on a 12-in. square filter-press gave the following results:*

Cake Thickness	*Filtering Time*	*Weight of Dry Cake*
1 *in.*	6 *min.*	1·9 *lb.*
2 „	21 „	4·0 „
3 „	46 „	6·1 „

It is planned to use plates giving a 3-ft. × 3-ft. filter-cake. A 1-in. cake will then weigh 18 lb., so the total cake thickness will be 125 in. (= D).

From the 1-in. and 3-in. results, $6 = A + B$; $46 = 3A + 9B$. Then $A = 1{\cdot}33$, $B = 4{\cdot}67$.

Checking for 2-in., $T = 2{\cdot}66 + 18{\cdot}68 = 21{\cdot}34$ min., and this is close enough to confirm the values for A and B. With two men working to discharge a press, the time will vary from 1 to 5 hr. per 1000 sq. ft. of filter area, according to the nature of the cake. As $CaCO_3$ is an easy material to handle, the lower figure may be taken. As each 3-ft. + 3-ft. plate represents 18 sq. ft. of filter area, it will take 1 min. to discharge ($E = 1$). Then the value of Q for most rapid working will be given by

$$Q = \sqrt[3]{\left(\frac{125}{9{\cdot}34}\right)} - \frac{1{\cdot}33}{28} = 2{\cdot}32$$

A cake thickness of $2\frac{1}{2}$ in. would be chosen, giving a filling time of 32 min. and an emptying time of 50 min. A 2-in. cake would give little difference, with 21 min. for filling and 67 min. for discharge, and would be better suited to a recessed-plate press.

The emptying time for a filter press is a rather doubtful quantity, resting on the manual dexterity of one or two workmen. It must also be remembered that emptying time is much more costly than

filling time; one man can look after two or three filter-presses while they are filling, but it is normal practice for two men to be employed on discharge. In view of these factors, results derived from equation (4) can only be treated as an indication of the cake thickness required and of the effect of alterations in the filtering constants and emptying time. Some practical figures for emptying times are given in the table below.

Filter-cake	Man-hours/1000 sq. ft. of Filter Surface	Size of Press
Tin Slimes . .	5·5	38 in. by 38 in.
Dyestuffs . .	5·0	22 „ „ 39 „
Dyestuffs . .	4·2	36 „ „ 36 „
Tungstic Oxide .	4·2	40 „ „ 45 „
Sewer Grease . .	1·8	36 „ „ 36 „
Sewage Sludge .	1·5	38 „ „ 38 „
Caustic Mud . .	1·0	42 „ „ 42 „

The limiting size for a filter-press plate is set by the load that two men can slide along the side bars, and it will be noted that the largest sizes mentioned above are about 42-in. square. It is also increasingly difficult, as the size of plate increases, to make a good liquid-tight joint round the large rims by pressure exerted from the end of a long press. The filter-cloth is not an ideal jointing material, and all filter-presses leak. It is best to erect each press inside a small compartment with watertight walls and floor, arranged for easy washing down. It has been found that a large central screw is usually the simplest and most reliable device for exerting pressure evenly round the rim of each plate, though corner screws, hydraulic pumps, and toggle actions of various types have been tried. The central screw may be very large, with a subsidiary ratchet wheel for easy hand operation. The ideal form of filter-press closure has not yet been discovered, though a very wide variation in methods has been applied.

The recessed-plate filter-press is cheaper but perhaps less widely used than the plate-and-frame press. In these, as shown in Fig. X–4, flat filtering plates, with corrugated surfaces, alternate with distance frames, which determine the cake thickness. These frames may be of any width, but are not normally less than 1 in., giving a sufficient thickness of metal for slurry inlet ports, as shown at A–A. The filter-cloths hang evenly over the surface of the plates, with no central port or ring distortion, although they are still called on, as a rule, to make the joint between plate and frame. As a result cakes of up to 4 in. or more in thickness may be built up in frames of

corresponding width, though the normal working range is from 1 to 3 in. The slurry is pumped along the inlet channel formed by the holes a, a in each plate and frame, and fills all frames simultaneously. It is common practice to provide air-vents on the top of each frame, discharging through a sight port into a return drain. This helps to avoid air-locks in the first stages of filtration. When discharge of slurry at the air-vents shows the frames to be full, these vents are closed, and the pressure then forces liquid through the filter-cloths and out at the liquid-discharge taps B, B. These are

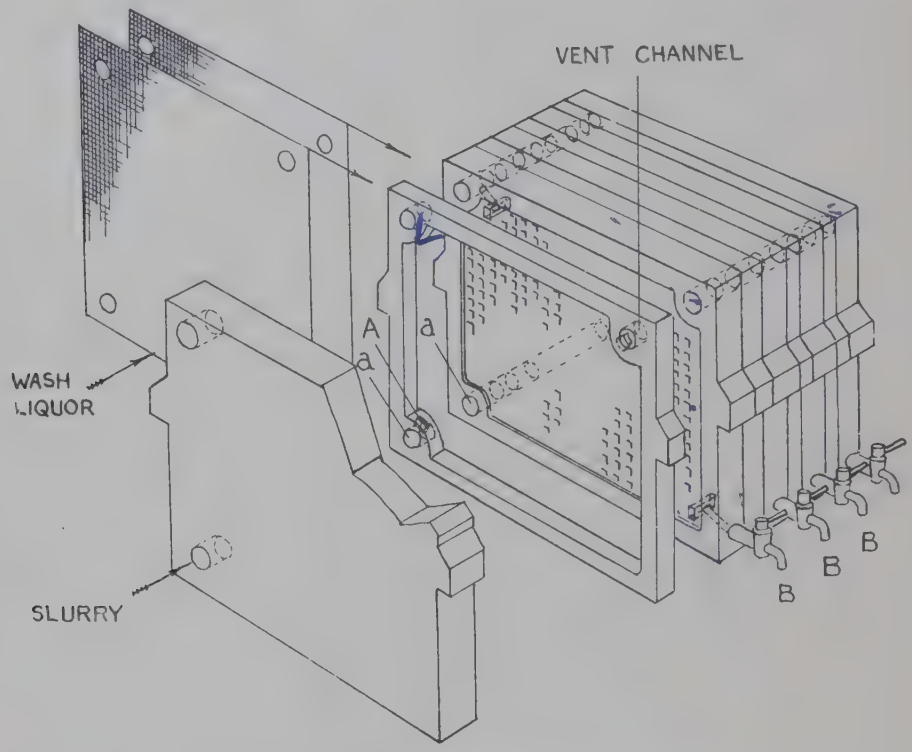


FIG. X–4.—PLATE-AND-FRAME FILTER-PRESS.

shown as discharging into an open channel, the most usual arrangement, but there is no difficulty in arranging for a closed discharge. In fact, the plate-and-frame design allows for a wide variation in the position of all fluid channels, and a number of alternative designs can be found in makers' catalogues. All types are alike, however, in providing for the so-called " through-washing " system shown in diagram in Fig. X–5. This also illustrates the filling of the chambers with slurry, followed by the building up and compacting of the solid cakes. The outlet taps on the special washing plates, which have a lead from the wash-water inlet to the filtrate channels, are then

closed and the wash pumped into the press. It enters the channels on the washing plates, flows through the cake and the double cloth, and out of the taps on the ordinary plates. These are set alternately with the washing plates, and it is usual to arrange for a numbering device to check this arrangement when the plates are assembled.

The washing part of a filter cycle is of considerable importance when the filtrate contains salts which must be recovered by concentration. The amount of wash-water must be as small as possible, and this can only be achieved by regular flow through all parts of the filter-cake at the same time. Ideally, an amount of wash-water

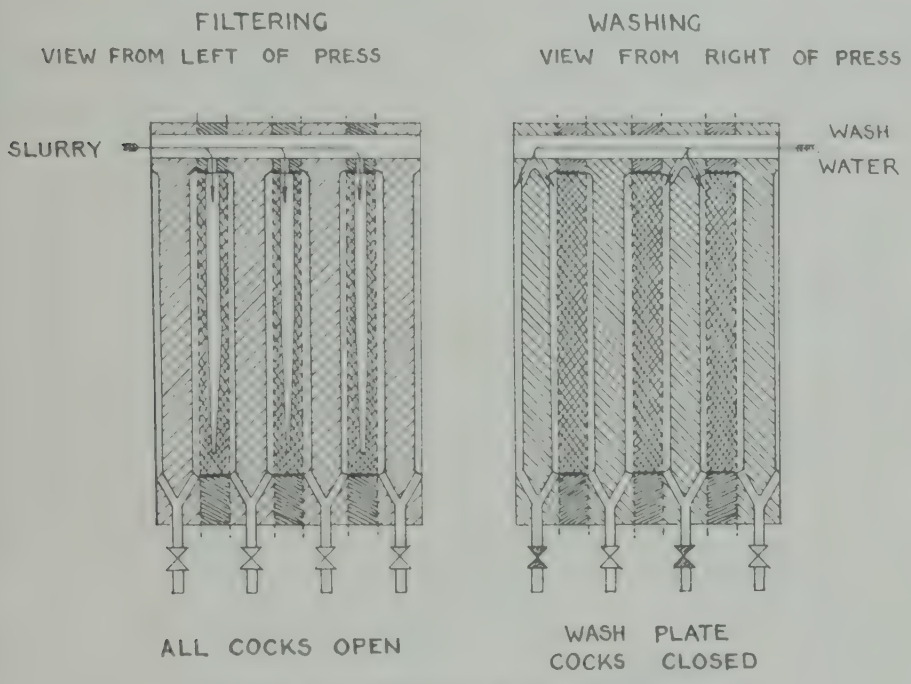


Fig. X–5.—Through-Washing Diagram.

equal in volume to the cake voids should displace all the original filtrate, and the salt concentration in the wash-liquor discharge should follow the heavy black line of Fig. X 6. This is never obtained in practice, but washing in a filter-press can give results approaching the line 1 as compared to line 2 for a pressure-leaf filter and line 3 for a rotary vacuum filter. Filter-press through-washing is therefore much better than washing 3 and nearly as good as 2. It is possible to incorporate through-washing into recessed-plate presses, but there is a much greater tendency to "short-circuiting" through zones of low resistance than in the plate-and-frame filter. Even in this latter there is a tendency for the cake to slip down in

the chamber, leaving a gap at the top. It has been claimed* that some improvement in washing is obtained by the use of flanged frames, but no manufacturer appears to incorporate this device as a standard fixture.

Washing rates cannot be based on the hypothesis of a steady cake resistance, as this tends to change with the removal of electrolytes, the formation of colloidal solids, and the setting-up of electro-osmotic potentials.† The rates may bear little resemblance to the original filtration rates if the wash-water follows a different channel to the original filtrate. In these circumstances it is desirable to utilise experimental figures, extrapolating between points on a graph of washing times *v.* cake thickness.

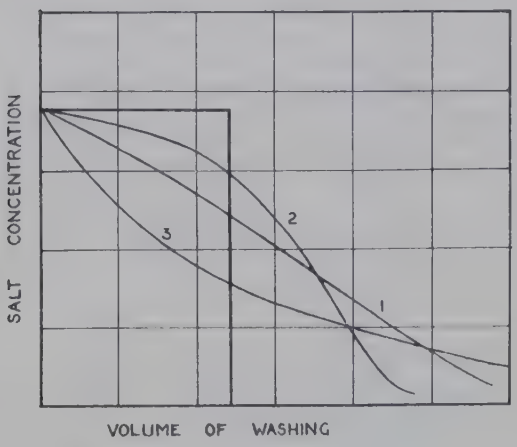


FIG. X–6.—WASHING CURVES.

Example 2. *In the conditions laid down for Example* 1 *above, it is required to wash the cake with three times its own volume of water. Experiment on the* 12*-in. square press gave the following figures:*

Cake Thickness	*Washing Time*	*Total Filling and Washing Time*
1 in.	3·0 min.	9·0 min.
2 ,,	6·1 ,,	27·1 ,,
3 ,,	8·9 ,,	54·9 ,,

From these the following table can be drawn up:

Cake Thickness	*Filling and Washing Time*	*Weight of 3-ft.×3-ft. Cake*	*No. of Cakes Required*	*Filtering Area*	*Emptying Time*	*Total Cycle Time*
1 in.	9·0 min.	18 lb.	124	2240	133 min.	142 min.
2 ,,	27·1 ,,	36 ,,	62	1120	67 ,,	94 ,,
2·5 ,,	39·5 ,,	45 ,,	52	930	56 ,,	96 ,,
3 ,,	54·9 ,,	54 ,,	41	740	45 ,,	100 ,,

On this basis, either a 2*-in. or a* $2\frac{1}{2}$*-in. cake would be chosen.*

* Alliott, *Proc. Chem. Eng. Grp.* (*Soc. Chem. Ind.*), 1936, 18, 68.
† Ruth, *Ind. Eng. Chem.*, 1946, 38, 564.

It must be pointed out again that this is not the most economical cake thickness, owing to the divergence of costs for filling and emptying. A series of figures given by Alliott* do not suggest that any definite relationship between filling and emptying times has been observed in industrial practice. It will be revealing to consider the incidence of labour charges and other costs on the examples given above.

Example 3. *A filter-press is required to hold* 1 *ton of* $CaCO_3$ *(dry weight) from the slurry as tested before, and the cake is to be washed with* 3 *volumes of water. The press can be built from* 2-*ft.* × 2-*ft. frames, discharged by* 1 *man at a rate of* 1 *min./frame, or of* 3-*ft.* × 3-*ft. frames, discharged by* 2 *men at a rate of* 1 *min./frame. Determine the most economical arrangement, neglecting capital charges, space rentals, and pumping charges.*

Experimental figures with the 12-*in. sq. press, extended to thicker cakes, gave the following results for the filling and washing times:*

Cake Thickness (in.) .	1	2	3	4	5
Time (min.) . .	9·0	27	55	92·5	141

The weight of dry solid in a 1-*in. cake will be:* (3-*ft.* × 3-*ft.*) 18 *lb.*, (2-*ft.* × 2-*ft.*) 8 *lb.*

Then the number of frames required for 2240 *lb. of dry cake will be:*

Cake Thickness (in.) .	1	2	3	4	5
2 *ft.* × 2 *ft. Frames*	280	140	93	70	56
3 *ft.* × 3 *ft. Frames*	124	62	41	31	25

As each frame will take 1 *min. to discharge (and reassemble) these figures represent also the emptying times. The relative costs are* 1 *for filling,* 3 *for emptying* 2-*ft.* × 2-*ft. frames, and* 6 *for emptying* 3-*ft.* × 3-*ft. frames. The following table shows the relative costs for the varying cake sizes:*

Cake Thickness	*Filling Time*	*Emptying Time*		*Cost per Cycle*	
		2 *ft.* × 2 *ft.*	3 *ft.* × 3 *ft.*	2 *ft.* × 2 *ft.*	3 *ft.* × 3 *ft.*
1 *in.*	9·0 *min.*	280 *min.*	124 *min.*	849	753
2 ,,	27·0 ,,	140 ,,	62 ,,	447	399
3 ,,	55·0 ,,	93 ,,	41 ,,	334	301
4 ,,	92·5 ,,	70 ,,	31 ,,	302	279
5 ,,	141 ,,	56 ,,	25 ,,	309	291

* *Loc. cit.*

A 4-in. cake would therefore represent the most economical thickness on both 2-ft. and 3-ft. presses. It would be necessary to use two presses for the 70 2-ft. × 2-ft. frames, so that on this basis a 3-ft. × 3-ft. press with 31 frames each 4 in. wide would be chosen.

The example has been greatly simplified by the omission of all but labour charges, but these are overwhelmingly important in a case of this kind. The change from 2·5 in. for most rapid throughput to 4 in. for most economical working illustrates the importance of economic factors in design.

If it is required to discharge a drier cake than can be obtained by simple pressure, air can be blown through the wash-water channels. This has the advantage of removing the wash liquor by displacement, and makes a further reduction in soluble solids as well as a reduction in moisture content.

The great simplicity of the filter-press and the ease with which it may be adapted for any type of filter-cake have made it the most widely used of all types of filtering plants for moderate loads. It is simple to arrange for heating or cooling of the entire press by making the plates hollow and using the space as a steam-heating chamber or for the circulation of chilled brine. The filter-cloths, with their dual function of filtering and jointing, are the least desirable feature. They are usually of wool for dilute acid liquors, or cotton for weak alkaline solutions. Metal cloths, from such wires as monel or stainless steel drawn thread, can be obtained, but they must be reinforced with softer material for the joint. Glass cloth makes a good filtering medium but cannot stand the stresses at the joint. Where any filter-cloth is rapidly attacked or clogged, the ease with which the filter-cloth can be changed in a press is a strong point in its favour, as compared to other types of filter. Cloths from synthetic resins, such as nylon and vinyl resin, are now being used for corrosive solutions, and in any case are good filtering media, as they do not normally swell when wetted. Rubber filter media—Multipore and Micropore—have been described but have not yet found wide application.

Probably the ideal way of feeding slurry to a press is by means of an air-pressure ram or " montejus ". This is a tank containing the slurry, to which air pressure is applied to force the fluid out. Obviously this can only be used for small-scale filtrations, but the method provides a steady pressure at the filter-cake and does not

break up fragile flocs of precipitate. The best type of pump is one which conforms as closely as possible to this mode of action, and suitable forms have already been described in the chapter on Pumping.

The varying effect of pressure on filter rates has already been mentioned. Even with relatively rigid cakes it may sometimes be desirable to form the first layers of cake at a relatively low pressure to ensure that they are not carried too far into the interstices of the cloth. Where the filter-cloth is very apt to block in this way, or where the solid has a very low critical pressure, a " filter aid " may be used. These can be regarded as ideal solids for filtration, chemically inert and physically stable. As a rule they are made from natural diatomaceous earth, the silica skeletons of prehistoric forms of life. A good filter aid is nearly pure silica, with individual particle structure of complicated highly porous form.* It has been found possible to prepare grades suitable for rapid filtration or for very clear filtrates.† In a typical case 1 lb. of filter aid 100 sq. ft. of filter surface was added to the first few gallons of a sugar solution, to give an optically clear filtrate. The cake of solid discharged from the press with a filter-aid backing resembling a sheet of blotting paper. Again, the addition of 1 lb. of a rapid filtration aid to 100 gal. of a 20% solids slurry gave a fivefold increase in filtration rate.‡

It may be mentioned briefly that several attempts have been made to " mechanise " the discharge of a filter-press, notably by means of a revolving central axis carrying knives or water sprays. These have not proved a success except in certain limited cases.

Pressure-Leaf Filters. The various forms of pressure-leaf filter represent an attempt to secure the flexibility and high operating pressure of the filter-press, while avoiding the multitudinous jointing system and heavy labour charges which seem inseparably linked with the older plant. Several forms of this type of filter have secured fairly wide adoption. One of the most successful, at any rate in the sugar industry, has been the Vallez filter. A series of circular filter-leaves, of the type shown in Fig. X 7, are threaded on to a stout central conduit, drilled with holes corresponding with each leaf. These leaves are built up from a disc of heavy crimped

* Salmon, *Proc. Chem. Eng. Grp.* (*Soc. Chem. Ind.*), 1938, 20, 20.
† Cummins, *Ind. Eng. Chem.*, 1942, 34, 403.
‡ Cogger and Merker, *Ind. Eng. Chem.*, 1941, 33, 1233.

wire cloth, covered by lighter wire on each side and then by filter-cloth bags, with joints at the central hub and periphery. The whole assembly of leaves is fixed in a cylindrical tank, formed in two semi-circular halves. The long joint between the halves is designed for permanent closure while the plant is working and is relatively simple to make liquid-tight. The central conduit projects beyond the ends of the tank through packing glands; one end is fitted with gearing which rotates the whole filtering device, while the other is connected with filtrate receivers. At the bottom of the lower half of the tank is a U-sectioned extension running the whole length of the filter-tank and housing a screw conveyor.

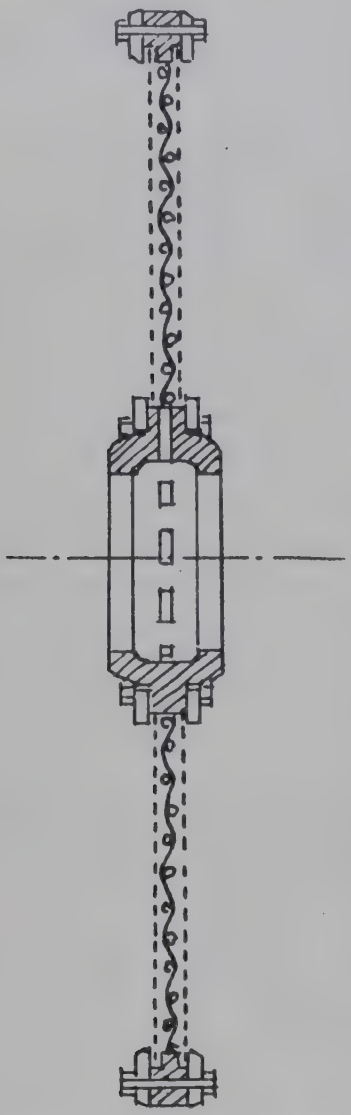


FIG. X–7.—VALLEZ FILTER-LEAF.

The operation of the filter follows lines common to all pressure-leaf filters, except that the filter-leaves rotate at 1–2 r.p.m., a special characteristic of the Vallez. With the press closed except for air-vents at the top of the casing, the slurry is pumped in until the tank is full. The vents are closed and pressure increased to any desired figure; filtration starts, the solid cake growing on the leaves while the filtrate is discharged through the central tube-axis. Growth in cake thickness is measured by the deflection of a light paddle pressing against the leaf surface and moving an indicator needle outside the cell. All slurries tend to settle, the coarser particles falling to the bottom of the tank, but this does not interfere with even filtration if the leaves rotate. When the cakes have reached a suitable thickness, the tank is drained under compressed-air pressure. This air pressure holds the solid in place, and blows some of the original filtrate out of the voids. Wash-water is then admitted slowly, displacing the air, and washing continued to any desired state. As it is possible that some shrinkage and cracking of the cake occur during the process of displacing the original slurry, it is usual to

mix some washed cake with the first washing put in—the "muddied wash-water" technique.

After washing the cakes can be partly dried by air-blowing before the air current is reversed, pressure being put on the inside of the filter-leaves. At the same time jets of water or scouring air sluice off the loosened cake and it falls into the conveyor trough. The conveyor carries the discharged cake out through centre or end doors according to the filter design. Inspection doors at the top of the filter allow the leaves to be inspected without opening up the tank, and if found satisfactory the filter is ready for another cycle.

As will be seen, the entire operation is conducted in a closed vessel, with a minimum of labour charge and spillage. Volatile

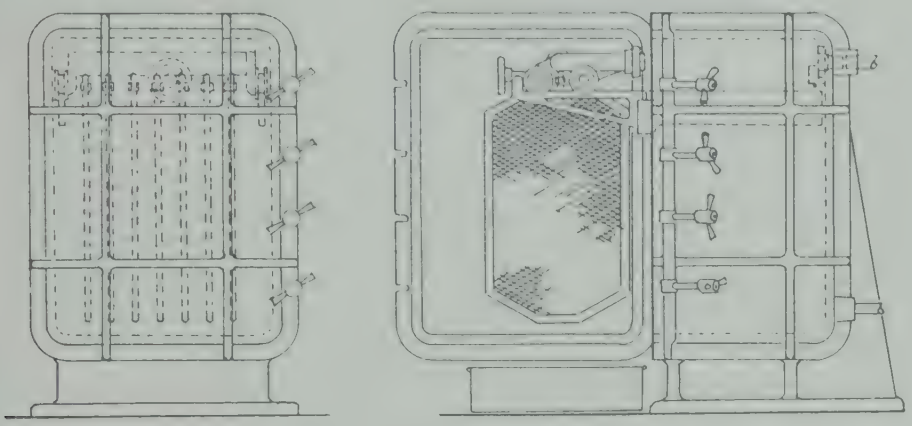


Fig. X–8.—Pressure-Leaf Filter.
(*Courtesy of Manlove, Alliott & Co.*)

liquids can be handled with ease, and insulation against heat loss is simple. At the same time, if the filter-cloths clog rapidly, changing is very difficult, involving the dismantling of the entire press, though this may be overcome in some other designs. A further point is that operation, as with the filter-press, is discontinuous and calls for careful supervision throughout. Attempts have been made to build continuous filters of the pressure-leaf type,* but they do not appear to have been too successful. It has been claimed,† however, that labour charges are less than half of those involved in filter-press working, even where leaf systems are used in which the cake is discharged by hand. A typical unit of this type is shown in Fig. X–8.

* *Ind. Eng. Chem.*, 1936, 28, 1065.
† Riegel, "Chemical Machinery".

In this, the "Easifilt", vertical leaves swung from a top outlet manifold are drawn out on rails to give easy discharge of the solid by scraping or hosing. Much larger filters of the same type have been put into operation in the United States. The Sweetland filter has a form similar to the Vallez, while in the Kelly the leaves form long flat plates, arranged as vertical curtains across a cylindrical tank opening at one end. In all pressure-leaf filters a large vessel has to stand a considerable internal pressure. This calls for heavy construction in a mechanically reliable material, and where corrosive materials have to be filtered this may not be a simple matter. Another point which must not be overlooked is that some chemicals,

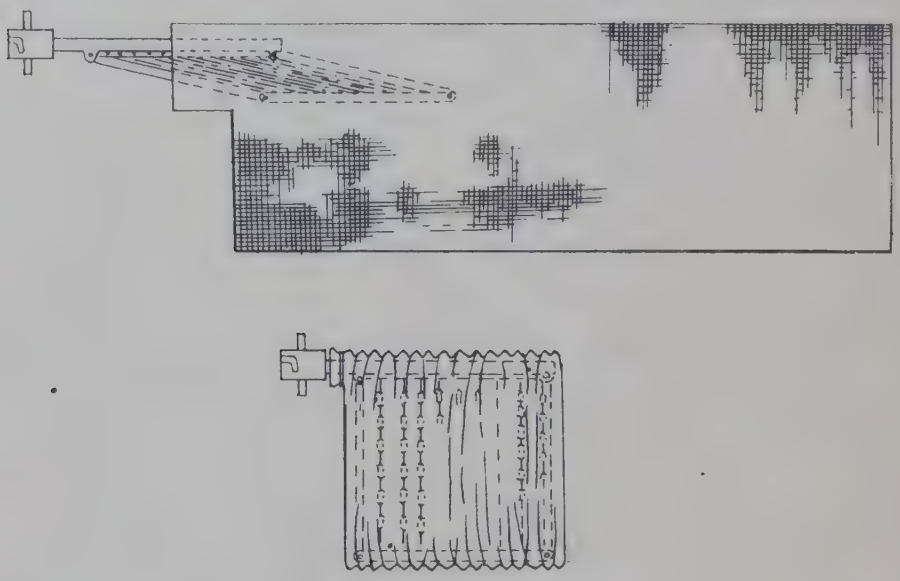


Fig. X–9.—Folded Filter.

such as delicate dyestuffs, are very sensitive to traces of metals, and the filter-press device of building in wood is not acceptable for pressure-leaf filters.

Analysis of the operation of a pressure-leaf filter follows the same lines as that of a filter-press. A compromise must be made between filling and emptying times for a batch of certain weight, while for the hand-discharged units the high cost of emptying must be allowed for. The amount of solid in a slurry fed to these leaf filters should be about the same as that for filter-presses—5% or less—but thicker mixtures can be handled if no rapid settling occurs. When the operation is one of clarifying, with a very small amount of solid, it is possible to crowd a great deal of filter surface into a comparatively small volume. An example of this is shown in Fig. X-9.

A filter-bag 9 ft. long by 3 ft. deep is 'crumpled' on to a frame 3 ft. by 3 ft., and a number of these are packed into a pressure tank, giving as much as 1000 sq. ft. of filter area in a volume of 50 cu. ft. Such a filter will be precoated with filter aid before operating on the liquor proper.

Rotary Vacuum Filters. Continuous filters of this type have steadily extended their range of application since the introduction of the first generally successful models about forty years ago. It is of interest to note that the difference between success and failure in these early units lay in the introduction of a raised wire winding

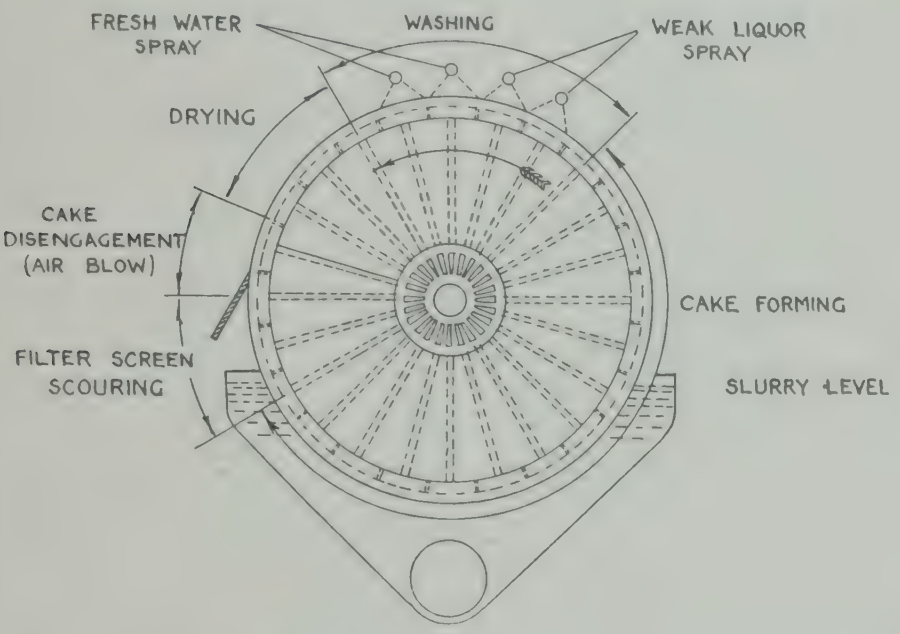


FIG. X-10.—ROTARY VACUUM FILTER.

which kept the knife scraper from touching the actual cloth. A wire winding is still a characteristic feature of most filters, and illustrates one of the principal difficulties met with in vacuum-filter operation. The usual filter consists of a drum, the periphery of which is divided into sections, as shown in Fig. X-10. Each of these sections is separately joined to a central valve system, which allows them to be connected to vacuum or to pressure services in turn. The drum surface is corrugated to form filtrate channels, and these are covered by a filter-cloth, held in place by wire winding, as mentioned above, or built up in sections, which are clamped into jointing grooves. As the cloth on a filter of this type is seldom changed, it is possible

to use special materials, such as a very fine-mesh metal wire cloth, but ordinary wool or cotton cloths are common.

The drum is partly immersed in a slurry tank and rotated slowly (from 1 to 0·01 r.p.m.). The sections under the slurry surface are connected to vacuum, and the filtrate sucked through the cloth, with solid building up a cake on the outside. The cake can then pass through a washing zone, a suction zone for drying, and an air discharge zone where pneumatic pressure under the cloth lifts the cake for a scraper to complete discharge. A typical valve arrangement for this is given in Fig. X–11, which shows a rotating plate carrying a ring of circular ports, one connecting to each filter segment, and the fixed covering plate which faces this. On the fixed plate, open-

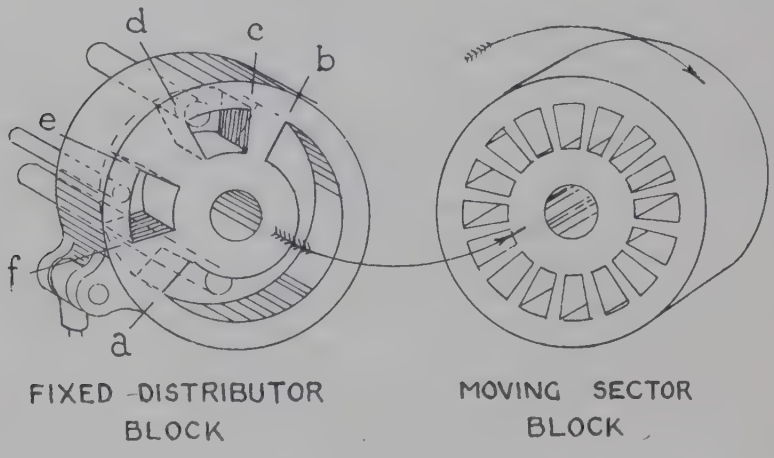


Fig. X–11.—Rotary Vacuum Filter Valves.

ing a–b is connected to the main filtrate receiver and vacuum, and covers the moving port openings leading to submerged filter segments. As each lifts from the slurry, it connects with zone c–d, and thence to wash receivers. Finally, each segment port passes to zone e–f, carrying air under pressure (about 5 lb./sq. in. ga.). There are a large number of variations of this cycle, with such modifications as reduced vacuum during cake formation, very long drying cycle, and cloth scouring after cake removal.

There are three difficulties commonly met with in working rotary vacuum filters. The first is the tendency of the slurry to settle in the trough, despite the general practice of pumping feed to a steady overflow. If efficient stirrers are put into the tank the cake is washed from the surface, while the slow rocking stirrers generally incorporated leave neutral zones in which settled solid builds up to a hard

mass. If very rapid settling is met with, it may be desirable to use the Dorrco filter, in which the filtering surface is inside the drum. These filters resemble a normal unit turned inside out, with the slurry carried in the tank formed between the partly closed ends. The cake is discharged by air pressure alone, and it has been found necessary to arrange for a pressure pulsation to give complete discharge. An alternative to the Dorrco filter, for rapid filtration of very coarse solid suspensions, such as crystals from a special crystalliser, is a top-feed unit of the kind shown in Fig. X–12. The feed flows directly on to the filter periphery from a head box, and a cake

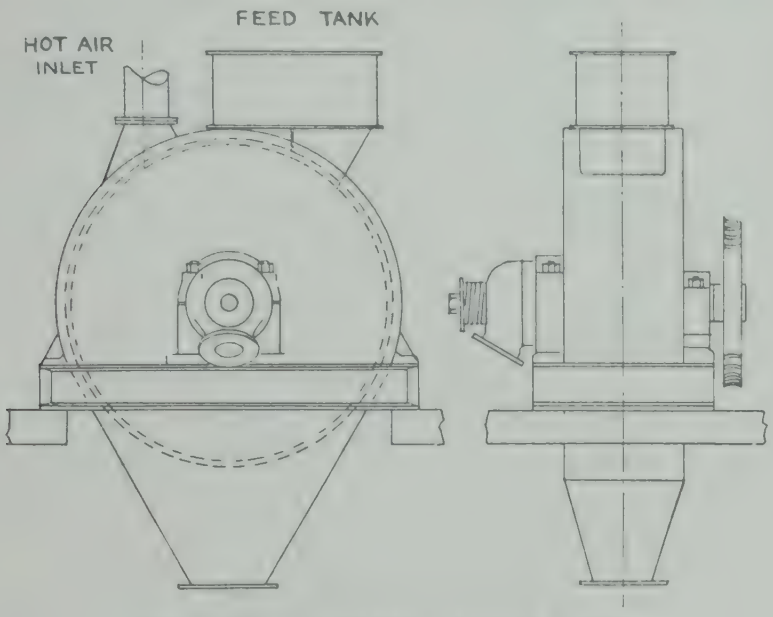


Fig. X–12.—Top-Feed Vacuum Filter.

is built up rapidly, to be held on during the rest of the cycle by a rapid flow of drying air. This gives a combination of filtering and drying which is growing in popularity. Radial extensions are now used to support the cake and allow thicker layers to be built up, and a double drum, with the feed entering the zone formed by the upper sections, has been described.*

The next operating difficulty met with in rotary vacuum practice is the washing, which is seldom efficient. As soon as the cake is lifted out of the slurry it starts to contract and crack, and attempts to correct this with heavy wash-water spraying either dilute the slurry or wash the cake off the filter. The cake may be compressed by an endless belt, spring-loaded to stretch over the surface of the

* Ramsey, *Chem. Met. Eng.*, 1944 (No. 7), 109.

filter and porous to allow for washing through it, while wash-water troughs may replace the normal sprays. Whatever the device employed, it is generally accepted that washing on these filters is poor, and it may be necessary to repulp the cake with wash-water and pass the resulting slurry to another filter.

The final difficulty lies in the cake discharge. It is not possible to discharge the cake by means of a sharp edge pressing directly on the cloth, as this would rub fine solid particles into the filter medium, and a very rapid falling off in filter rates would ensue. The introduction of wire winding, with the discharge blade resting on the wires, made a vital improvement, but the blocking of the cloth can still be serious. It is now customary to use a rubber rather than a metal edge for discharge, and to use a pulsating air-flow in the discharge air lines. A porous rubber diaphragm has been suggested* as a mount for the filter-cloth; this would contract under vacuum and dilate sharply, disintegrating the cake, when the discharge zone is entered. Finally, and this seems to be the most successful expedient, the filtering surface is covered by an endless perforated band, which leaves the drum after passing the drying section and carries the cake with it. A small runner bends the band sharply and the cake breaks away without need for scraping, especially where the band is formed from a series of strings. The arrangement, together with the smoothing band for washing, is shown in Fig. X–13, and has the added advantage that the pressure-air section of the cycle is dispensed with.

A more recent development has been the precoating of the filter with a thick layer, up to half an inch in thickness, of filter aid. The normal filter-cake is built on top of this, and a sharp discharge knife shaves off the cake with a thin layer—as little as 0·001 in.—of the filter aid. The knife is geared to travel inwards at a very low speed, peeling off a continuous layer of the precoat, but giving steady working for a long period before the precoat is down to a dangerous level. As an interesting example of mechanised control it may be noted that a patent has been taken out for the knife travel to be governed by a photo-electric cell responding to the whiteness of the fresh filter-aid surface revealed at each rotation. The precoat filter is adapted for clarifying liquids as well as for handling large bulk solid cakes.

The output of a filter will vary widely with the slurry being

* Cullen *et al.*, *Trans. Inst. Chem. Eng.*, 1934, 12, 210.

treated, but if this is fixed, the cake thickness will depend upon actual filtering time. This filtering time is equal to the time of revolution of the drum multiplied by the fraction of the periphery immersed. The degree of immersion will vary from 75% with filtrations giving a wet cake and a maximum of clarification volume, down to 15% with the specialised top-feed units. For normal operation, with washing and drying, a submergence of 30–40% is customary. The operating choice lies between the extremes of a thin cake built up quickly and a thick cake built up slowly. In a filter of surface S the total cake area discharged in unit time will be

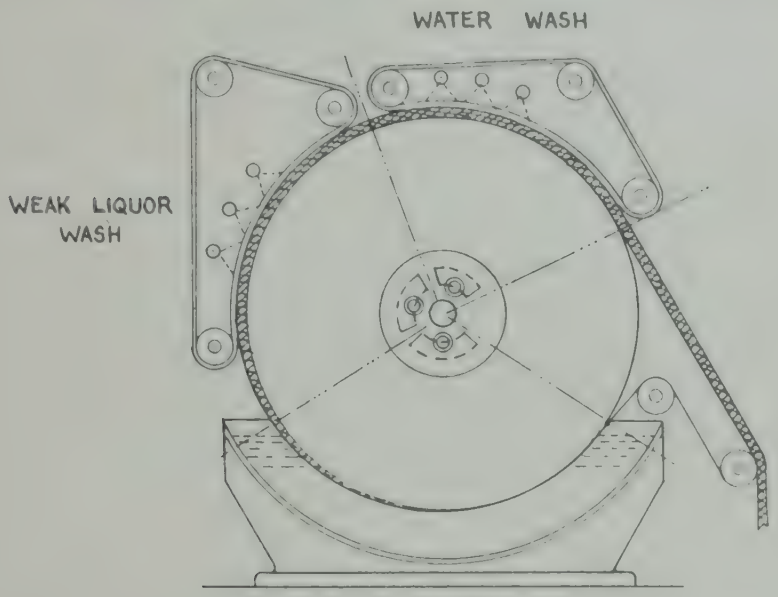


Fig. X–13.—Cake-Pressing Belts and String Discharge.

S/θ where θ is the time of one revolution. If the thickness of the cake is given by Q, it may be assumed that $\theta = AQ + BQ^2$, or, more approximately, $\theta = BQ^2$. Then the volume of cake discharged in unit time will be SQ/θ or SQ/BQ^2, which reduces to S/BQ. That is to say, the output will vary inversely as the cake thickness. The maximum output from any filter will thus be obtained by running with the thinnest possible cake, and limiting factors are set by the dryness, degree of washing, and the need for a cake which is firm enough for proper discharge. In practice it has been found that a scraper discharge calls for at least 6 mm. ($\frac{1}{4}$ in.) of cake thickness, though it has been suggested that cakes as thin as 1 mm. can be handled by a string discharge. It is always necessary to make laboratory tests on any particular slurry, and fortunately the actual

cake formation on a rotary vacuum filter can be closely simulated by a submerged filter-leaf of quite small size, working under the same vacuum as is proposed for the full-scale unit.

Example 4. *A filter is required to produce* 500 *lb./hr. of dry cake from a given slurry. Samples tested in the laboratory, on a filter-leaf of* 0·1 *sq. ft. area, under a suction of* 15 *in. Hg, gave the following results:*

Time (1)	*Cake Thickness*	*Moisture Content after draining for period equal to* (1)	*Weight of Wet Cake*
10 *sec.*	4 *mm.*	—	—
20 „	6 „	32	0·12 *lb.*
30 „	8 „	—	—
60 „	12 „	—	—
90 „	15·5 „	28	0·31 „

It may thus be assumed that the cake will contain 30% *of moisture, and will weigh* 60 *lb./cu. ft. A thickness of* 8 *mm. will be arbitrarily chosen, giving a time of formation of* 30 *sec. The weight of* 1 *sq. ft. of cake will be* 1·58 *lb., containing* 1·11 *lb. of dry solid. A total cake area of* 450 *sq. ft. will be required each hour. Suppose a filter of length* L *and diameter* D. *Then the cake area per revolution is* πDL. *If the immersed surface is one third of the whole, the time of rotation must be* 90 *sec., or* 40 *revs./hr.*

Then $40 \times \pi DL = 450$, *whence* $DL = 3{\cdot}6$.

As the general tendency in rotary filter practice is to make the diameter up to 3–4 *ft., a filter* 1 *ft. long by* 3 *ft.* 8 *in. diam. would be chosen. The total filtering area is* $11\frac{1}{2}$ *sq. ft., and the output of dry solid is* 43·5 *lb./sq. ft./hr. If it had been deemed possible to work with a* 4*-mm. cake, the filter area would be reduced to* 7·5 *sq. ft., and the output per sq. ft./hr. would increase to* 66·5 *lb. However, the speed is already on the high side.*

The free-filtering slurries usually handled in rotary vacuum filters give filter rates from 0·25 to 5 gal./sq. ft./hr., while the cake weight ranges from 10 to 150 lb./sq. ft./hr. The wide range with somewhat similar slurries shows the need for experiment in every case. The vacuum pumps must handle at least 1 cu. ft. of free air/min./sq. ft. of filter area, and for crystalline cakes in a top-feed filter up to 100 cu. ft. of capacity is required. Power requirements for

rotation of the filter are small, a 100 sq. ft. unit taking only 2 h.p. A typical layout for filtration, with concentrated and weak liquor receivers, is shown in Fig. X–14. All the plant is at floor level, but a barometric leg separator traps entrained liquor, while pumps remove the filtrate. It is possible to erect the filter well above the ground and discharge all filtrates by these barometric legs, which, for very thin cakes, can act as vacuum pumps.

The construction of a filter is complicated, which means that

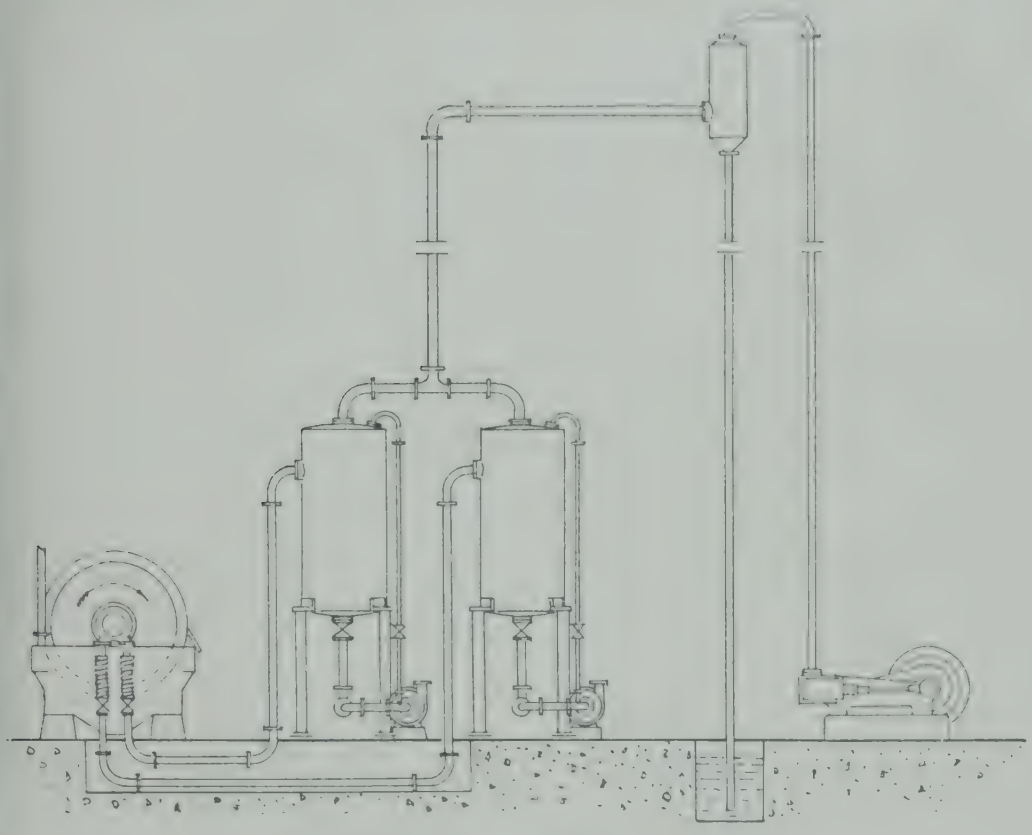


Fig. X–14.—Rotary Filter Layout.

reliable materials must be used. It is possible to use wood, but the structure is rather fragile and susceptible to bacterial decay. Lead-lined filters are possible, but are apt to wear at critical points, and stainless-steel construction is expensive. A filter in porcelain, made in Germany about 1939, cost some £500 for a laboratory model of 3 sq. ft. filter area. Corrosive solutions cannot therefore be handled so easily as in filter-presses, and the typical materials for construction of a rotary vacuum filter are cast iron and mild steel. As mentioned above, the filter-cloth is often in fine metal gauze—down to 300 meshes/inch—backed by a coarser material.

A rotary vacuum filter with a filter area of 500 sq. ft. will occupy a floor area of about 25 ft. by 20 ft. This is a greater space than would be required for a filter-press of similar area—about 25 ft. by 6 ft.—and pressure-leaf filters are even more economical in floor space. In an effort to improve this " spread " of plant, the vacuum disc filter was developed. This unit, as shown in Fig. X–15, has a very similar construction to the Vallez pressure leaf, in that the filter-leaves are built up on a central hollow shaft. A pressure difference, in this case formed by a vacuum inside the leaves, builds up a cake as the filter units rotate. Operation is in the open, however, and only a portion of the leaf is immersed, with segments connected separately to vacuum or pressure, as with the ordinary rotary vacuum filter. The cake must be discharged between the various leaves, and the operation is a little more complicated than

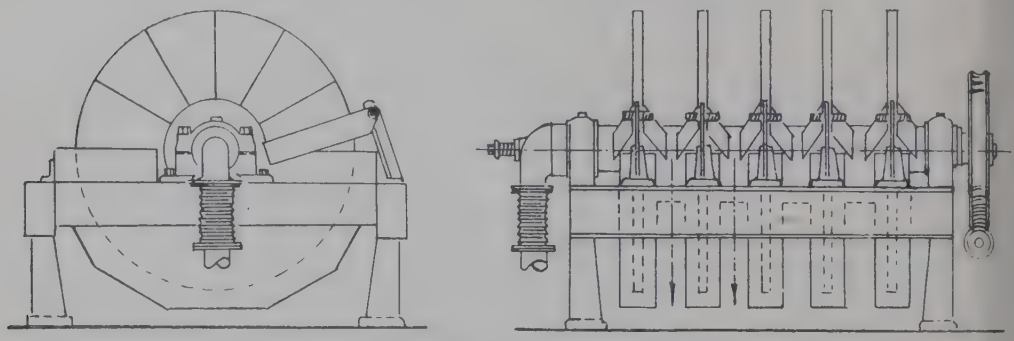


FIG. X–15.—ROTARY VACUUM DISC FILTER.

with a simple drum, while washing problems again arise. As a result the disc filter has not found very wide application, despite the fact that the floor space per unit of filter area is about half that of the drum type.

The simple vacuum filter tank, or " nutsch " filter, resembling a large Buchner funnel, in which suction is applied to a space below a perforated floor, is still used for strong acid filtrations, especially where small quantities of material are handled. Vacuum leaf filters of the Moore or Butters type, in which banks of filter-leaves are lowered into tanks of slurry, are now almost obsolete.

A development of the rotary filter drum which makes use of fluid head to carry out coarse filtering, or straining, is found in the rotary strainer shown in Fig. X–16. The drum is covered by a fine wire screen and is closed at one end, being attached at the other to a feed tank which keeps the drum two-thirds full of water. The drum

is rotated slowly –peripheral speed 3 ft./min. –by a small motor, and the fine silt cake on the screen lifts out of the water, to be washed by subsidiary jets into a waste trough. This is reminiscent of the discharge arrangements of the Dorrco vacuum filter. The wire cloth forming the screen should be fine enough to remove all particles of more than 50 microns, but coarse enough to clean easily with water sprays. Ordinarily 200-mesh wire cloth has an aperture of 76 microns, and can be plated up to a wire thickness which gives a 50-micron aperture. These rotary filters can handle very large volumes of water with a small loss of head. For example, three 7 ft. 6 in. diam. filters passed 1,000,000 gal./hr. of river water.*

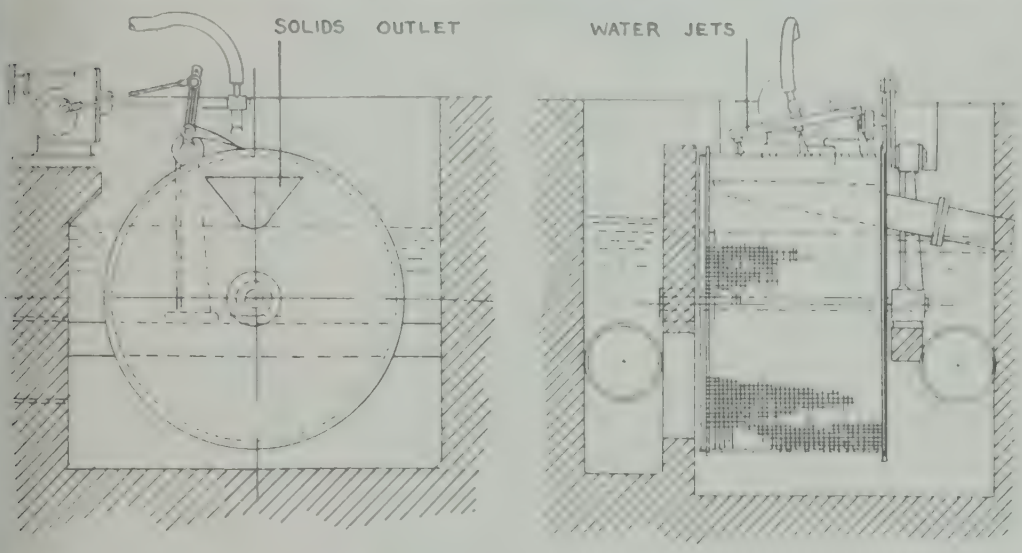


Fig. X–16.—Water-Strainer.

For final clarification of the water from these filters, it is still general practice to use large sand or gravel filter beds. These are too specialised to describe in this book, and have been mainly developed in connection with bacterial changes and town water treatment. When a small quantity of sterile water is required for chemical work, the simplest type of filter available is some type of porous earthenware thimble. These can be obtained with a wide range of porosity –from 5 to 200 microns –and have lately been supplemented by porous metal products.

Edge Filters. The last important class of filter is the "edge" or "stream-line" filter. These are essentially clarification filters, giving optically clear filtrates from a slurry containing small

* Boucher, *The Engineer*, 1942, 173, 420.

quantities of solid, and can be used for sterilising. In their simplest form they consist of piles of paper or metal discs with a central hole. When the pile is clamped together under pressure, a central channel is formed, which can be connected to a common header carrying several sets of discs. The header is built into the lid of a closed vessel holding the feed liquid, and this is put under fluid pressure, as in the case of the pressure-leaf filter. Clear liquid is forced into the inner channel, leaving the solids as a cake on the outer edges of the

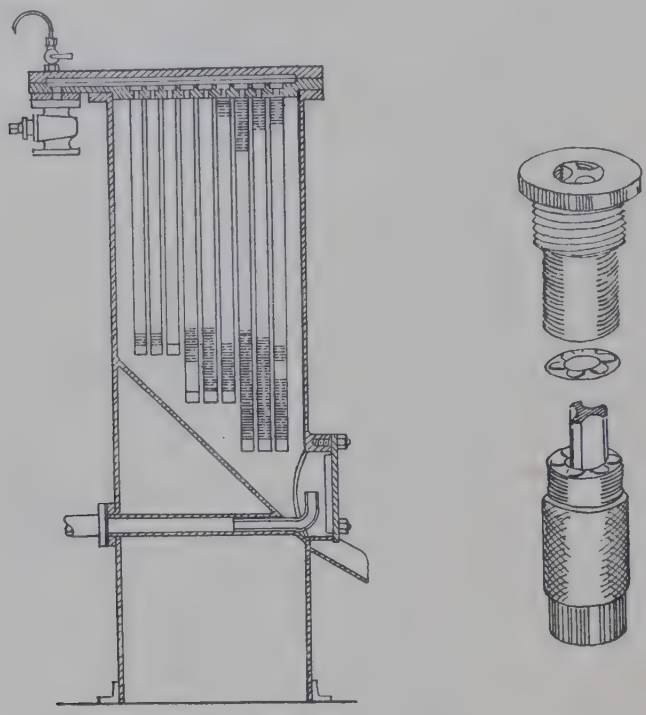


FIG. X–17.—EDGE FILTER.
(Courtesy of Metafiltration, Co.)

discs. Developments from this simple form have led to the use of filter aid as a precoat on the discs, which are now formed from metal, with embossing to give a fixed space between each ring and the next. A typical filter of this type is shown in Fig. X–17. These filters can be used in the clarification of viscous liquids, as well as water and similar fluids. The most popular application of this is in the clarification of used lubricating oil. Output is low, but the very fine colloidal fouling agents are efficiently removed. When the resistance of the deposited solids builds up to a predetermined figure, a short back-pumping period breaks off filter aid and solids together and they can be removed as a thick sludge. Other applications are

found in the clarification of varnishes, plating solutions, and all kinds of beverages. In this last the removal of bacteria is of special importance. Rates of filtration under a pressure of 10 lb./sq. in. vary from 0·5 gal./sq. ft./hr. for lubricating oil up to 100 gal./sq. ft./hr. for water sterilisation. There are no moving parts in these filters, and they are easy to construct in acid-resisting materials. It would appear that the edge filter, in its various proprietary forms, is replacing older types of clarifying filter based on recessed-plate filter-press design.

CHAPTER XI

CENTRIFUGAL PRACTICE

The filters described in the preceding paragraphs have handled all types of solid–liquid slurry reasonably well. The cake discharged has usually contained 10–30% of moisture, even when the drying period was considerable. Also, when coarse solids, of about 0·01 in. size, are being filtered, the cake build-up is uneven. If solids of low moisture content are particularly required, or if large solid particles are to be freed from water, some type of centrifugal separator is commonly employed. The field of centrifugal operation is much wider than this, however, and a review of the other functions of a centrifugal plant is given below.

If a weight of 1 lb. is spinning in a circle of diameter D with a linear velocity V, the effective force acting outwards in a radial direction is equivalent to $2V^2/gD$ lb. When this linear velocity is given by a speed of N revs./sec., $V = \pi DN$ and the effective weight may be written as $2(\pi DN)^2/gD$, or $2\pi^2N^2D/g$, or, in F.P.S. units, $0{\cdot}613N^2D$. This multiplication of the effective weight is defined as the Centrifugal Effect, and is a measure of the increase in settling or draining rate over simple gravity effect.

For a 36-in. centrifugal basket, revolving at 20 revs./sec. or 1200 r.p.m., the value of the centrifugal effect is $0{\cdot}613 \times 20^2 \times 3$, or 736. This is the maximum value, as the various masses making up the basket and load all rotate on different diameters. The typical centrifugal basket has the bulk of its weight contained in the outer wall, and is never filled right up. It can be shown that, for liquid loads, the mass exerts force outwards as though the entire mass was concentrated at the rim. In these circumstances it is desirable to base centrifugal calculations on the assumption that the total rotating weight is, in fact, moving on the outermost periphery of the basket.

Consider a basket of diameter D and length (depth) L, weighing with its load a total weight of M pounds. Then the weight acting outwards will be $0{\cdot}613N^2DM$. The surface against which this

weight is forced will be πDL, and it follows that the resulting pressure will be $0{\cdot}613N^2DM\ \pi DL$ or $0{\cdot}195N^2M\ L$ lb./sq. ft. This pressure may be very considerable. In a 36-in basket a wall of water 4 in. thick and 1 ft. deep ($L = 1$) will weigh normally 174 lb. Then at 1200 r.p.m. ($N = 20$) the pressure will be $0{\cdot}195 \times 20^2 \times 174$ or 13,570 lb. sq. ft. (94·2 lb. sq. in.), to which must be added the stress due to the weight of the basket rim.* The basket must be strong enough to resist these stresses, and although the problem of design is not serious, especially with a good constructional material, the centrifugal in operation is equivalent to a vessel under internal pressure, and must be treated with proper respect.

The load which can conveniently be introduced into a centrifugal basket is proportional to the cross-sectional area, or to D^2. At the same time the stress caused by a unit weight of load will be proportional to $V^2\ D$. The area available to resist this load will be proportional to D and hence the stress produced in the basket shell per unit of load will be $V^2\ D^2$. As the total load will be determined by D^2, the stress will be given by some function of $(V^2\ D^2) \times D^2$ or V^2. That is to say, the ultimate stress on a centrifugal basket will be proportional only to the peripheral speed. But the centrifugal effect is determined by V^2/D, and therefore, for the highest centrifugal effects compatible with safe loads, the diameter of the centrifugal basket must be as small as possible.

The centrifugal separator may be designed

(*a*) to remove water from a solid–liquid slurry, using a perforated basket—centrifugal drying.

(*b*) to separate two, or even three, phases of different density —centrifugal separation.

(*c*) to increase the settling rates of solid particles in a fluid—centrifugal clarification and classification.

The last two functions usually require a non-perforated basket.

The standard batch centrifugal used for drying in the chemical industry has a cylindrical basket, with depth less than diameter, made with perforated walls and some type of false bottom. This basket is driven from a vertical shaft attached to a motor directly overhead. The whole centrifuge swings freely from the top bearing and is self-balancing, a factor which makes it necessary to charge the basket evenly. In normal practice this is done by running slurry into the basket while the centrifuge is rotating at a moderate speed.

The force developed is sufficient to form a slurry wall against the filtering medium which lines the basket. The filtering medium may be built up from a coarse wire screen directly against the wall of the basket, followed by a filter-cloth of cotton, wool, or metal wire fabric. When charging is complete, the speed of rotation is increased rapidly to full operating value, and filtrate is torn from the solid surfaces by centrifugal force and passes through filter medium and basket wall out into the casing surrounding the centrifugal basket.

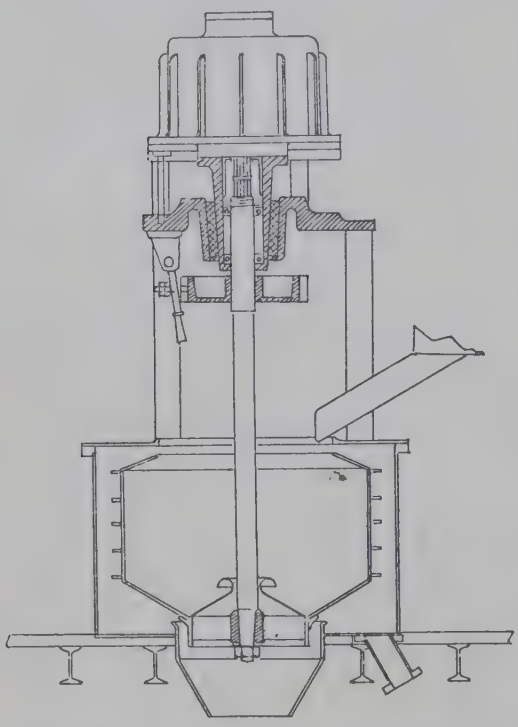


FIG. XI–1.—OVERDRIVEN CENTRIFUGAL DRYER.

As will be seen below, the speed of liquid removal can be very high. The initial drying period can be followed by washing, the wash liquor being sprayed on to the moving solid wall. When the solid has been re-dried, the basket is slowed down and stopped by some form of braking — often the longest section of the whole cycle— and the solids are then raked down by hand and dropped through openings exposed in the false bottom. The general arrangement of an "overdriven" centrifugal of this type is shown in Fig. XI–1. The thin wall of the basket is reinforced by rings, but these are perhaps less common than a stouter and self-sustaining wall. Machines of this type are rotated at speeds which give a centrifugal effect of 1000–1200, even with baskets of 42 in. diameter. Speeds have been steadily increasing over the last twenty years, with parallel calls for higher power and for exact control of operation.

The output from any centrifugal dryer is determined by the time necessary to reach a certain predetermined dryness. This point is illustrated by Fig. XI–2, which shows the sharp fall in output from a machine when the allowable moisture content is steadily reduced. It is probable that for any solid cake the moisture content decreases steadily with time of "spinning" towards a basic minimum, determined by the physical characteristics of the material and the

magnitude of the centrifugal force, but no general formulae are available. The cycle of operations will therefore be determined by the material being handled. In a sugar factory, where centrifugal operation has perhaps been most carefully studied, a cycle might be as follows:

Charging	60 sec.
Acceleration	60 ,,
Drying	30 ,,
Washing	30 ,,
Drying	30 ,,
Braking	60 ,,
Discharging	90 ,,
Total Cycle Time	6 min.

A 36-in. basket, 24 in. deep, will hold about 4 cwt. of sugar, so that the output of sugar, of 1–2% moisture content, is 2 tons/hr. A machine of this size will not take up more than 30 sq. ft. of floor area, and it has been claimed that centrifugals are more economical in floor space than any other filtering unit of equal capacity. The principal merit of the plant, however, is the low moisture content, particularly valuable with soluble solids that are required in as pure a form as possible. It is easy, also, to build a centrifugal basket in corrosion-resistant material, and rubber- or ebonite-covered units are common.

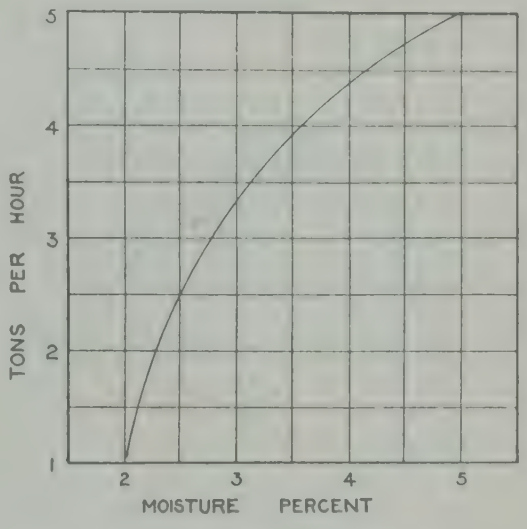


FIG. XI–2.—OUTPUT OF CENTRIFUGAL DRYER.

The principal disadvantage appears to be the need for manual control and discharge. It is possible to arrange for an automatic control, but the cycle is complicated, and it has generally been found necessary to allot one process hand to each two machines. Automatic unloading devices can be used, such as ploughs which cut into the cake as the basket rotates and shave off the solid in layers. A stop prevents the plough from disturbing the filter medium. Wear and tear on these ploughs is considerable, as a hard crystalline cake

may have been compacted at pressures up to 500 lb./sq. in. A suggestion was made some years ago that the discharge edge should be vibrated electrically, but no report of the practice has been noted. These automatic unloading devices have been more popular with horizontal-spindle machines, as described below.

The over-driven type of centrifugal has the great advantage of clear discharge from the bottom, but calls for a fair amount of headroom. This may be accentuated if a battery of the machines are driven from a common shafting. The power required for driving a large centrifugal is always high, but starting and stopping mean big variations. A 42-in. machine takes 75 h.p. in acceleration but

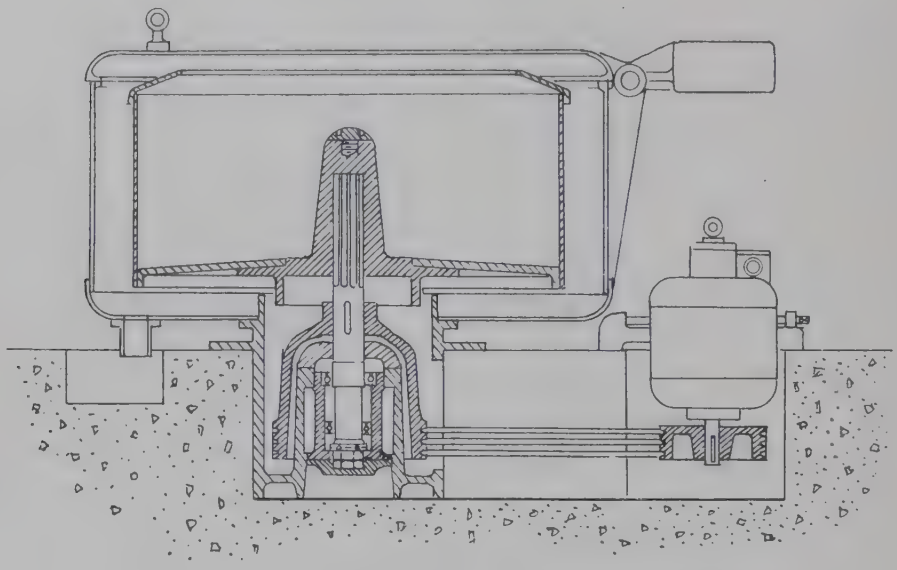


Fig. XI–3.—Under-driven Centrifugal Dryer.

30 h.p. when running at full speed, and it is possible to drive six machines off a common shaft by the same total installed power that would be required for three machines with separate motors. At the same time belt drives in chemical plant suffer badly from corrosive atmospheres, and are usually avoided where single machines are being operated.

The under-driven centrifugal, as shown in Fig. XI–3, has not been so popular in the chemical industry as in the laundry and textile drying processes, but has been applied in certain special processes. The most notable is the nitrating centrifuge for the manufacture of guncotton. In this centrifuge the casing is water-cooled and is first filled with nitrating acid; the cotton is added slowly to the

perforated pan, which is, of course, also full of acid, while it revolves slowly to give mixing and heat transfer. When nitration is complete, the acid is drained from the casing and the basket revolved at full speed to remove the bulk of the adherent acid from the gun-cotton. This is then discharged by hand to the drenching tanks. When under-driven centrifugals are used for drying textiles, the centrifugal forces are not so high as in chemical plant practice, but the baskets are bigger—up to 7 ft. diameter. Loading and discharging are by hand, and the basket can only be put in motion when a top lid is firmly closed. The drying is controlled by an

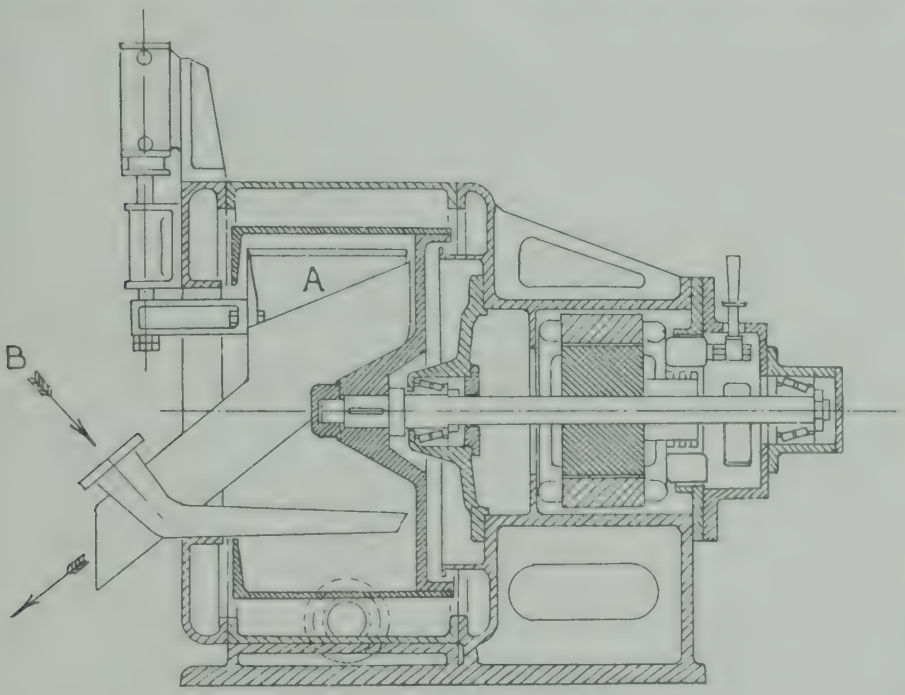


Fig. XI–4.—Horizontal-Axis Centrifugal Dryer.

automatic timer, which stops the centrifugal after a specific time. The baskets with their contents are lifted out by travelling cranes and inverted for discharge. A 6-ft. diameter machine, running at 580 r.p.m., takes up to 25 h.p. for operation.

The requirements of mechanical discharge appear to be leading to a wider adoption of horizontal-spindle machines, which lend themselves to this form of operation. In the plant shown in Fig. XI–4 the basket is carried as an extension of the axis of an electric motor and runs at full speed throughout the entire cycle. The charge is fed in through the spout B, usually as rapidly as possible; this is claimed to give an ideal build-up of solid in the cake, with the

coarsest particles in the outer layers. After washing and drying, the travelling knife A is set in motion and slices out the cake. The movement of the knife is exactly controlled, and the edge stops at 1 mm. from the filter-cloth. The whole cycle time may be less than 2 min. with a crystalline solid, such as sulphate of ammonia. At the same time large baskets can be used, of up to 80 in. diameter, while maintaining speeds of over 700 r.p.m. A centrifugal of this type 80 in. diam. by 36 in. deep can dry a charge of sodium bicarbonate once every eight minutes, while a similar charge of ammonium sulphate can be worked through in two minutes. A smaller machine

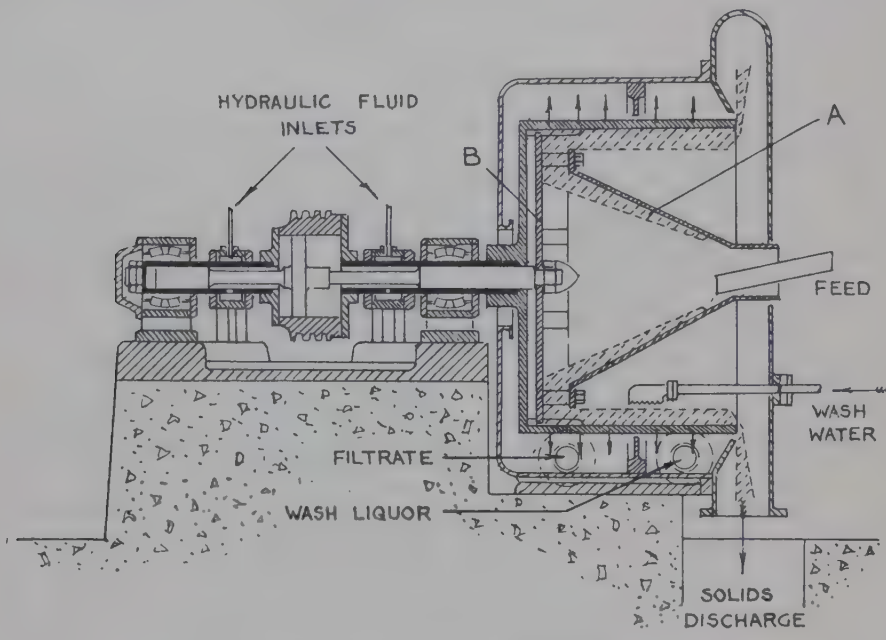


Fig. XI–5.—Continuous Centrifugal Dryer.

of the same construction, 20 in. diam. and rotating at 1800 r.p.m., will give an output of 2·5 tons of NaCl/hr., while working on a one-minute cycle. Power requirements vary from 50 h.p. for a 60-in. machine to 15 h.p. for a 20-in. unit, but all these centrifuges take a heavy overload during charging periods.

Although these machines will keep up a steady discharge of dried solid, operation is essentially by batches, and a varying power load must result. A nearer approach to truly continuous working is obtained by the Escher-Wyss centrifugal. A diagram of this unit is shown in Fig. XI–5; the cake is built up continuously from slurry fed through the funnel A, which rotates at a sufficient rate to spread the feed evenly. The bottom of the basket is formed by the

ram B, which moves to and fro inside the perforated wall over a short distance (0·5 to 1 in.) with a frequency of 5 to 30 strokes/min. The deposited solids are forced along the wall at each forward stroke, and the gap left as the ram recedes is filled with fresh slurry. The power requirements are uniform and low, a plant with an output of 2 tons/hr. of crystals taking only 3·5 h.p. for driving, with 3 h.p. for the ram. This centrifugal was widely used in Germany,* and the quality of the product was improved by the introduction of a dry air current at the outlet edge of the rotor. An alternative form of continuous centrifugal consists of two concentric baskets, the inner being in the form of a screw conveyor and travelling at a slightly different rate from the outer perforated shell. Solid is conveyed slowly through the machine by the spiral while drying, washing, and redrying are carried out in the various zones of travel. It is difficult to arrange for thorough drying in such machines and they do not appear to be very popular. A possible use is found in the de-watering of small coal from a washery, in which process a 36-in. machine can handle 30 tons/hr. of coal of $\frac{1}{8}$ in. and below in size.

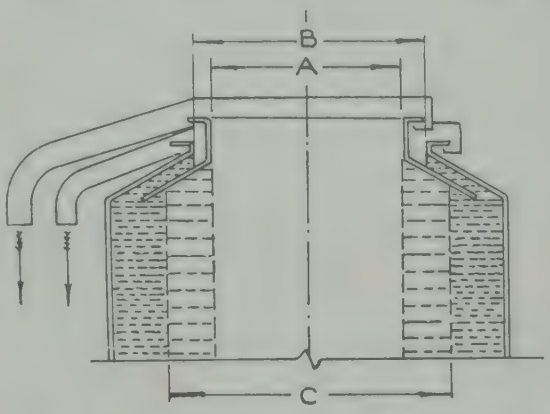


FIG. XI–6.—LIQUID–LIQUID SEPARATION IN CENTRIFUGE.

Centrifugal Separation. When a mixture of two liquids or a suspension of solid particles in liquid is fed into a non-perforated centrifugal bowl, the settling effects of gravity are accentuated and the mixture will split up into a heavy and a light ring, with the former on the outside. If the original mixture is fed in at one end of a bowl and arrangements made for divided overflow at the other, then steady streams of two phases may be drawn off. The system of discharge at the top of a vertical bowl, fed at the base, is shown in Fig. XI–6. This shows a section through the bowl, with the two rings of liquid meeting along a diameter C. A submerged weir allows the heavy liquid to pass under it and thence to an outlet on diameter B, while the light liquid overflows at the top on a diameter A. Then a heavy seal of depth (C—B)/2 balances a light

* *B.I.O.S. Rept.* 1587.

seal of depth (C—A)/2 and as long as this seal is maintained the relative amounts of each phase flowing through the machine are not important.

The time taken for the original mixture to separate into two distinct layers of the kind suggested in Fig. XI–6 will determine the output of these machines. In a typical liquid suspension the droplets will be of all sizes down to 0·001 mm., and will settle at rates determined by Stokes' Law if undisturbed. A liquid drop of

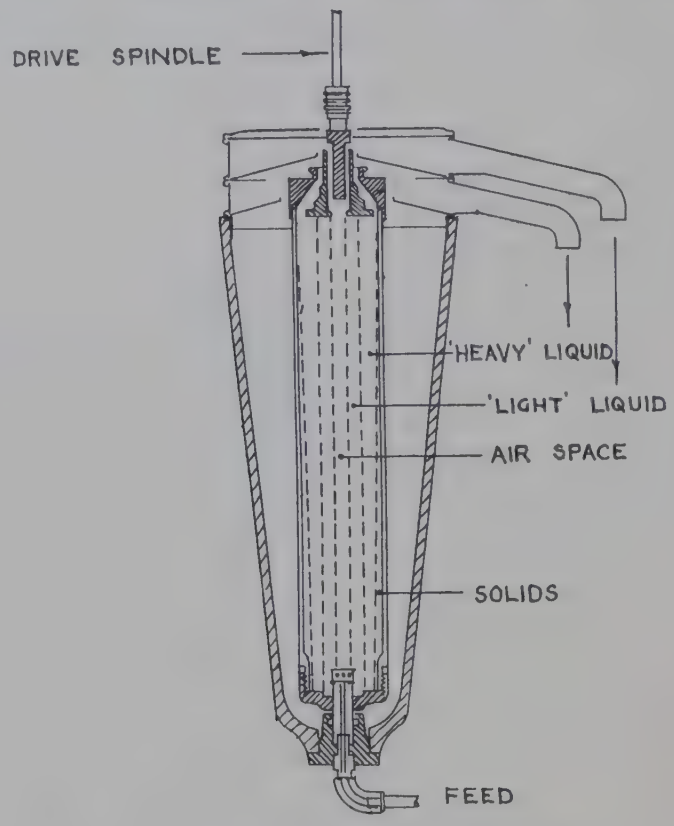


FIG. XI–7.—SHARPLES CENTRIFUGAL SEPARATOR.
(Courtesy of Sharples Centrifugal, Co.)

sp. gr. 1·2 and diam. 0·01 mm. will settle in water at about 0·5 mm./min.; the effect of centrifugal force on this settling rate is to multiply by the centrifugal effect, which in the case of certain separators may be 10,000. The settling velocity becomes 8·3 cm./sec., if Stokes' Law still applies. It is certain that considerable interference by other droplets will affect this settling, and in practice it is not possible to calculate the time required to separate any given mixture. The point will be taken up again when dealing with size separation, and at this stage it is only necessary to indicate

that a very great increase in settling rate can be obtained, determinable only by experiment.

The typical liquid separator is made up either as a long narrow bowl unit or in the form of a broad bowl with baffles to break up liquid swirl and provide channels for the settling phases. The former type has been developed by the Sharples organisation, and a "Super Centrifuge" of standard form is shown in Fig. XI–7. The long narrow unperforated rotor is 4 in. in diameter and 30 in. long, freely suspended and driven from the top. The mixture is fed in at the bottom of the bowl, which rotates at speeds up to 20,000 r.p.m., at rates sufficient to give two clear liquid discharges at the top. If required, the same machine will separate traces of solid, which are retained in the bowl as a cake and discharged periodically by stopping and lifting the whole bowl from its casing. In the cleaning of wet and dirty lubricating oil, a Super-Centrifuge gave 80 gal. of water-free oil per hour, with about 5 gal. of water and sufficient solid for an hourly discharge. By running a suspension through the centrifuge at a high speed, large solid fragments could be removed to leave the finer dispersion unchanged, and this has been turned to account in the preparation of fine enamels and paints.

The larger-diameter bowl, with baffles, has found widest application in the separation of cream from milk, but is met with in similar work on chemical plant. The bowl, as shown in Fig. XI–8, contains a series of concentric cones, which break up the chamber into a series of discs of 5 to 10 mm. in depth. The liquid fills all these disc spaces and the whole bowl rotates at speeds sufficient to give centrifugal effects of about 5000. As the spaces slope down and out, the heavier liquids pass outwards against the outer walls, forcing the lighter component inwards and up towards the centre. If only one baffle were used, each individual droplet in separation would need to travel a considerable distance, but the narrow disc spaces allow a rapid formation of both liquid phases, as suggested in the diagram of Fig. XI–9. The bowl arrangement can be modified to give a clarifying action, just as with the Super-Centrifuge, while adjustment for varying liquid densities can be made by altering the weir overflows. It is possible to arrange for the continuous separation of two liquid phases and the slower but still continuous discharge of a heavy sludge. In an operation of this kind on fish oil, a dry oil was separated from water and traces of silt at a rate of 660 gal./hr. in a 24-in. machine, taking 3·5 h.p. for driving.

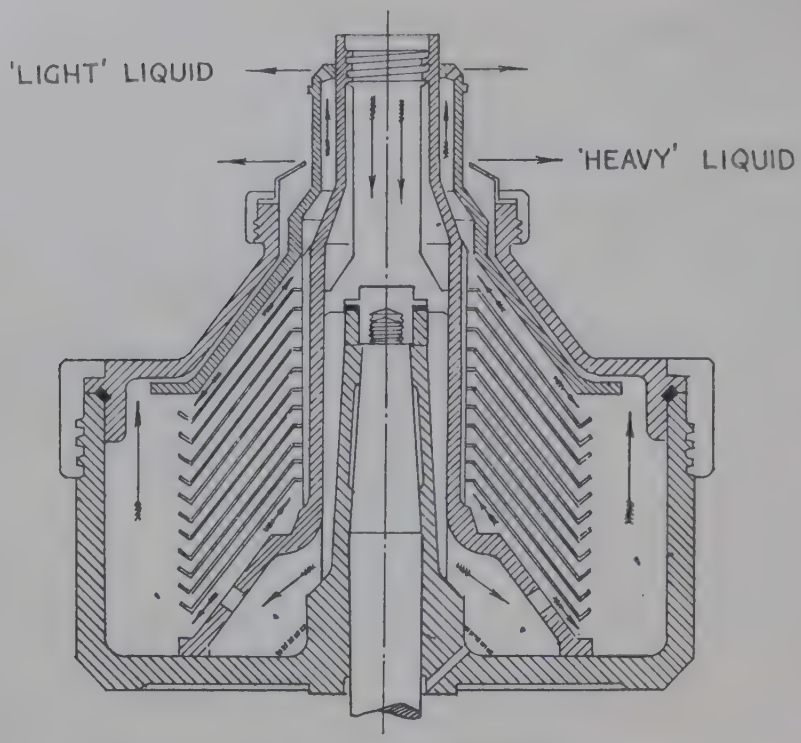


FIG. XI–8.—ALFA-LAVAL CENTRIFUGAL SEPARATOR.
(Courtesy of Alfa-Laval, Co.)

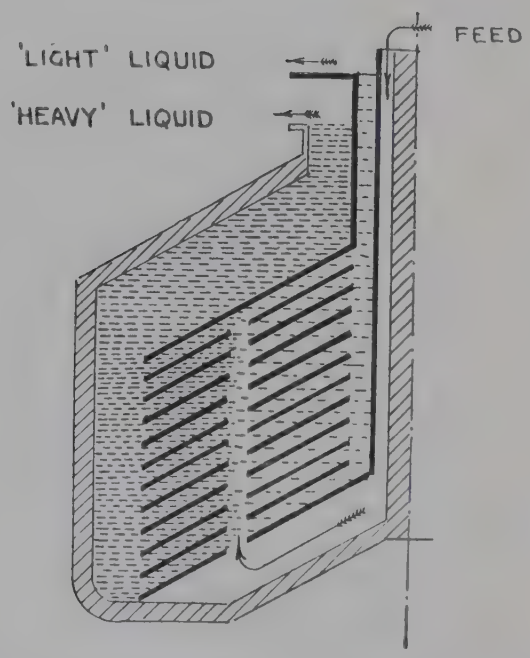


FIG. XI–9.—SEPARATION IN MULTI-CONE BOWL.

When one liquid discharge still contains solid in suspension on discharge from the centrifugal, it is clear that the machine has acted as a classifier. The rates of settlement of all but the finest particles in a suspension are such that the centrifugal would serve only in the separation of very finely divided solids. For this reason the centrifugal has had little direct application in classification of liquid slurries. As will be seen in the later chapter on Size Separation, applications of the centrifugal effect are to be found principally in the separation of air suspensions of solids. When a centrifuge is used directly, the particles discharged without settlement are usually below 0·015 mm. in size, but little published information is available.*

* But see Lyons and Johnson, *Amer. Inst. Min. Met. Tech. Publ.*, 2195, 1947.

CHAPTER XII

GAS CLEANING

The removal of dusts and mists from gases is a more difficult process than the removal of a vapour. Dust-laden producer gas bubbled through caustic soda solution loses all carbon dioxide, but still holds the bulk of the dust. The gas envelope surrounding small particles is difficult to break down, while many mists carry electrostatic charges. It is vital in many chemical processes to remove these particles as completely as possible, while dust-laden waste gases should not be discharged into the atmosphere. The particles held in suspension in normal gas currents will be those which have " settling velocities " of less than 1 ft./sec. The problem of settling velocities will be dealt with in the chapter on Size Separation, and it may simply be noted here that all particles settling in air have an ultimate falling velocity which will not be exceeded, no matter how far the particle may fall. The following ultimate falling velocities have been observed for water droplets in air:

Diam. in mm. .	0·1	0·07	0·04	0·01	0·001
Rate in ft./sec. .	0·87	0·43	0·15	0·008	0·00008

The normal dust or mist particle will therefore be below 0·1 mm. in size and may be assumed to have a mean settling velocity of about 0·01 ft./sec.

A simple settling chamber is of little use in removing these dusts. Even if it were possible to give regular streamline flow in a long conduit, a 50-ft. length of 6-in. main would be required to settle out all particles of above 0·01 mm. from a gas stream moving at 1 ft./sec. A large proportion of the finer dust would remain, and the total volume of gas treated would only be 700 cu. ft./hr. In order to increase this settling velocity recourse is made to centrifugal plant. If a stream of dust-laden air is forced into a circular path, the swirling motion sets the dust particles in motion outwards across the gas stream at a rate which is normally equal to the product of centrifugal effect and normal settling velocity.

The cyclone dust collector in its simplest form is a cone with a cylindrical top section, into which the gas stream enters tangentially at velocities of the order of 100 ft./sec. The centrifugal movement forces the dust particles to the sides of the cyclone, and they slide down towards the cone bottom as shown in Fig. XII–1. If the diameter at the top of the cone is 3 ft., and the linear velocity of the inlet air is 100 ft. sec., a centrifugal effect of $(100^2/1{\cdot}5 \times 32{\cdot}2)$ or 207 can be obtained. The velocity of a 0·01-mm. water droplet will become 1·66 ft./sec. outwards, and a mean time of 1 sec. in the cyclone should be sufficient to remove all these droplets if a steady centrifugal force is obtained. As the volume of a cylinder 2 ft.

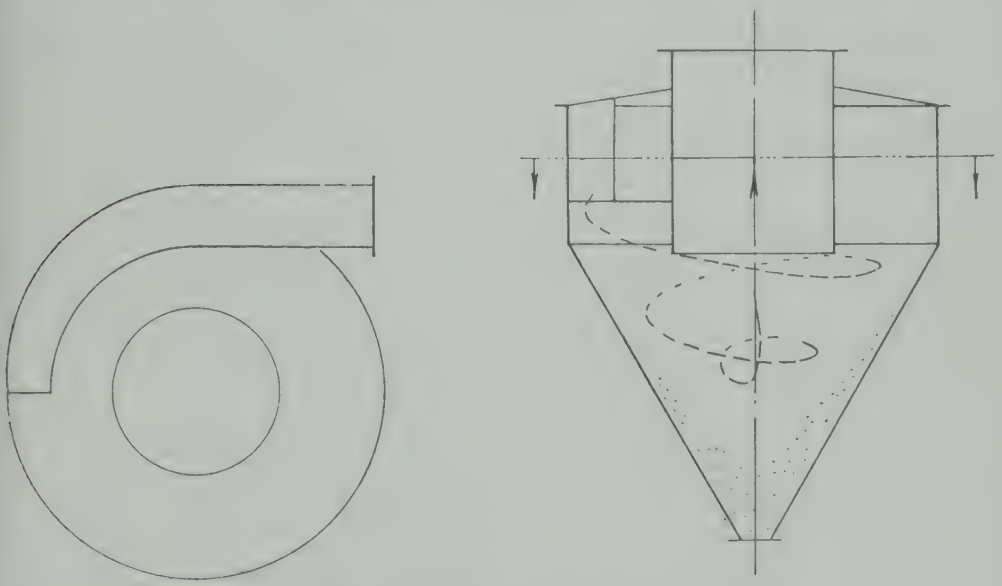


Fig. XII–1.—Cyclone Dust Collector.

deep by 3 ft. diam. is 14 cu. ft., an approximate capacity for this cyclone will be 14 × 3600 or 50,000 cu. ft./hr. of air. The entrance conduit to give an inlet tangential velocity of 100 ft./sec. must be 0·14 sq. ft., or a slit 1 ft. long by 1·68 in. wide. The pressure drop across the cyclone, assuming all the velocity head to be dissipated in eddies, cannot be less than 2·5 in. of water gauge, representing a power consumption of 0·3 h.p. Actual power figures are found to be from three to four times as much as this, because of incidental losses.

The design of a cyclone is much more complicated than in the simple outline given above. It has been found that, in practice, movement of the gas in a cyclone takes either a double spiral track

(the most general case) or a double eddy path, as shown in Fig. XII-2a and 2b.* The double spiral path is followed with all short broad cyclone extractors, and is only converted into the double eddy system by an elongated cylinder and cone, having the tangential inlet rather lower than usual on the cylinder wall. In the form shown in Fig. XII–2b a dust-collecting pocket is set on the outer edge of the two eddies, and this dust-skimming device is a common

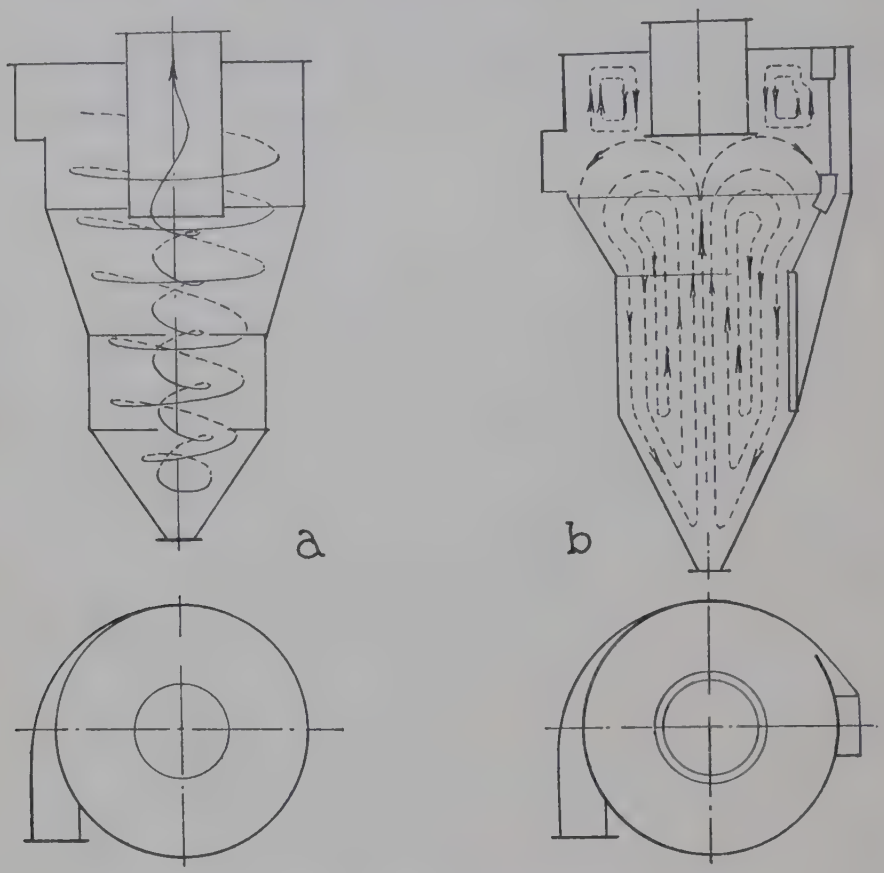


FIG. XII–2.—CYCLONE SEPARATORS FOR FINE DUSTS.

accessory to several recently developed forms of cyclone. To reduce pressure drop a system of radial vanes in the inlet duct has been proposed, but it does not seem to have found much application. It is an interesting fact that the pressure drop for a given air-entrance velocity drops as the dust content increases from zero up to about 35 grains/cu. ft., a maximum decrease of about 20% of the original figure having been noted.† Where the amount of gas passing

* *Ind. Chemist*, 1942, 18, 433 and 477 ; 1943, 19, 25.
† *Trans. Amer. Inst. Chem. Eng.*, 1946, 42, 511.

through a cyclone extractor varies, it is desirable to alter the inlet aperture so as to obtain a constant air velocity. In the course of a very full discussion* on this type of plant, H. L. Larcombe has suggested the reduction of cyclone design to standard forms, and embodied his ideas in a series of charts, which show capacity plotted against size and pressure drop for ordinary and for high-performance cyclones. However they may be constructed, these cyclone separators are recognised as relatively inefficient for the removal of any particles of below 0·005 mm. in size, as will be seen from the performance chart of Fig. XII–3. It may be possible to increase this efficiency by the use of a number of small cyclones, only 6 in. diameter or less, instead of one large unit. The effect of these small separators is to increase the ratio of surface to volume, and in so doing they may check re-entrainment of the dust. This is a fairly general cause of low efficiency, and may also be checked by running a current of water down the cyclone sides. The water flow is not always effective, owing to the difficulty of wetting the dust. Special plants have been built for this purpose, with a spinning disc on to which a stream of dry dust and a stream of water flow simultaneously.

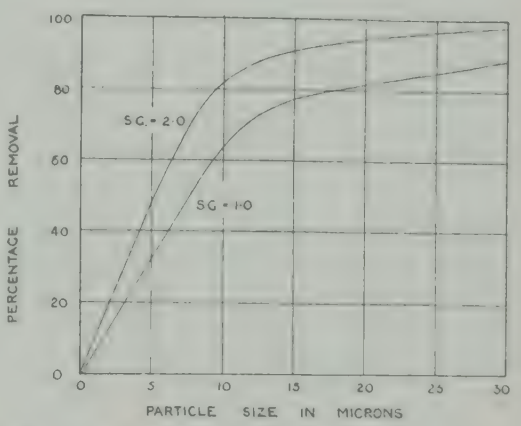


Fig. XII–3.—Cyclone Performance.

The gas swirl of the cyclone may be reinforced by water sprays, the droplets of which pick up the dust particles and are thrown out of suspension as a sludge. This is an enlargement of the idea of using wetted surfaces to remove dusts by impingement. When a current of gas carrying dust or mist particles in suspension turns rapidly through a right angle, as shown in Fig. XII–4, the particles are acted on by momentum and centrifugal force, and tend to strike against the deflecting surface. In an analysis of this type of scrubber G. L. Fairs† suggests that the size of the smallest particle that can be separated from a gas stream by this means will vary inversely as the square root of the pressure drop and directly as the clearance

* *Ind. Chemist*, 1942, 18, 433 and 477; 1943, 19, 25.

† *Trans. Inst. Chem. Eng.*, 1944, 22, 110.

distance between the plates—D in Fig. XII–4. This is the basis of the impact-plate scrubber, known in the acid industry as the Calder-Fox and in the gas industry as the P. and A. scrubber. Originally a number of perforated or slit plates were used, with the holes on each

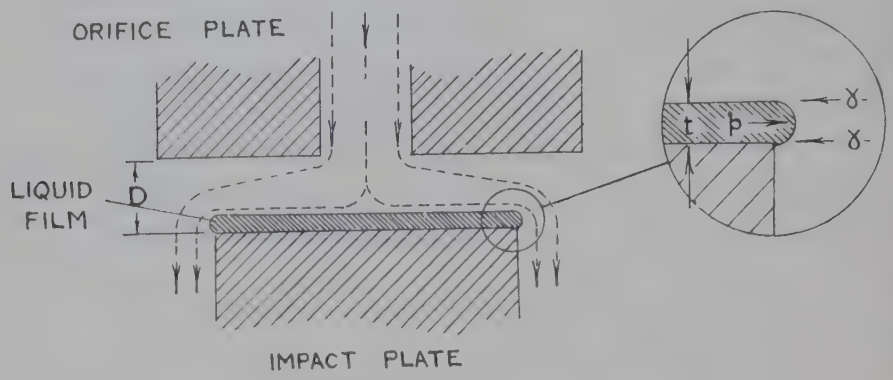


Fig. XII–4.—Impact Scrubber Operation.
(G. L. Fairs, *Trans. Inst. Chem. Eng.*, 1944, 22, 110.)

plate staggered to those of the next. Now it is more usual to rely on two or three plates, and to set these more closely. A modern arrangement from the sulphuric-acid industry is shown in Fig. XII–5; the orifices are slits formed from glass bars set closely

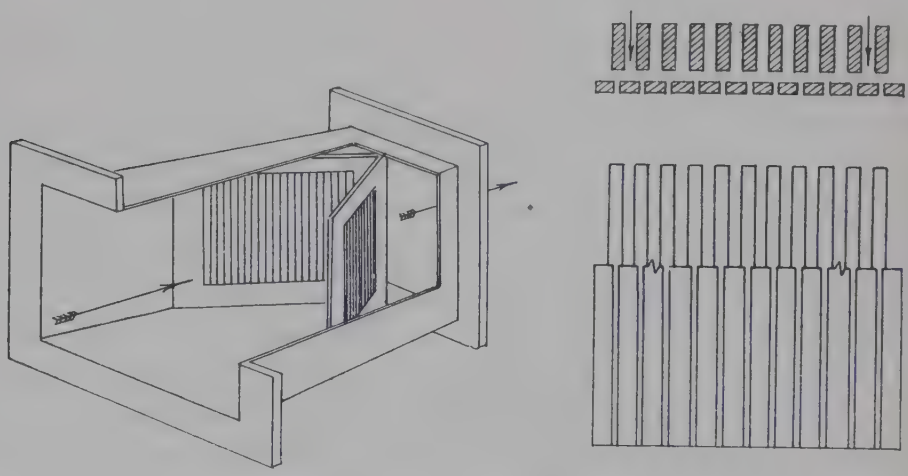


Fig. XII–5.—Calder-Fox Precipitator.

together, and will remove droplets of acid down to 0·002 mm. in size when slits of $\frac{1}{32}$ in., with a similar clearance, are used. The pressure drop is high for such efficiency and may reach 10 in. of water gauge. There is also some re-entrainment of acid from the edges of the slits, and only the physical properties of the sulphuric

acid film (Fig. XII–4) prevent this from being more serious. It is probable that the maximum velocity of air through such scrubbers will be some 100 ft./sec. Impact scrubbers can only be used for a freely flowing liquid dispersed in mist form, and are no use for dusts, though some attempts have been made to simulate mist deposition by running a stream of liquid over the impact plates. A special form of impact drum, which rotates and washes off the precipitate in a tank below, has also been built.*

Small air-filtering plants are available in which a single impact at high velocity is replaced by a number of possible impacts as the gas pursues a serpentine course through a filter pad of wires coated with a viscous oil. These filters can be made up from special wire cloths and washed out periodically, or made from some cheap fibre and simply discarded when the accumulated dust has checked free air-flow. A unit of this type will handle up to 500 cu. ft./min./sq. ft. of filter area, with a pressure drop of about 5 mm. water gauge. Such a low pressure drop suggests that the filter relies on chance eddy currents to remove the finer dusts. When absolute freedom from dust is required, filter slabs of slag wool can be used, but the filter rates are much slower—7 to 9 cu. ft./min./sq. ft. Normal compressed air always contains traces of oil from the compressor and is filtered free from this and other impurities by porous filters of the same type but of lower porosity. The pressure drop is greater, but this is not so serious as with gases at atmospheric pressures. If sterile air is required, then a porous earthenware filter can be used. A proprietary filter of this type ("Aerox") can be obtained with pore sizes ranging from 0·2 to 0·005 mm., the finest size giving air rates of 3 cu. ft./sec./sq. ft. with a pressure drop of 25 lb./sq. in.

A novel form of dust collector is shown in Fig. XII–6. The dust-laden air enters a cone-shaped collecting vessel, moulded from a slotted sheet through which the air is discharged. Heavy particles are collected in the ordinary "classic" way simply by the reversal of the air flow, the air being discharged through the slots in a direction opposite to the inlet flow. The distinguishing feature of this design, however, is that the cone and slots are formed in such a way that the air flow at the inside of the cone is made to undulate with high frequency oscillations, thereby inducing an aerodynamic force directed away from the filtering surface. This force keeps dust particles, even down to a very small size, afloat in the air

* B.P. 562,394.

flow, carrying them as a suspension continuously increasing in concentration to the cone apex, from whence they pass to a small cyclone. Here the bulk of the dust is deposited and the gas returns to the main stream.

Any liquid-spraying device will produce a large amount of wet surface for dusts and mists to deposit on, and subsequent removal of the relatively large droplets is easy. Spraying droplets, falling across the gas stream in any form of conduit, will give practically no additional pressure drop, and if all dusts were easily wetted

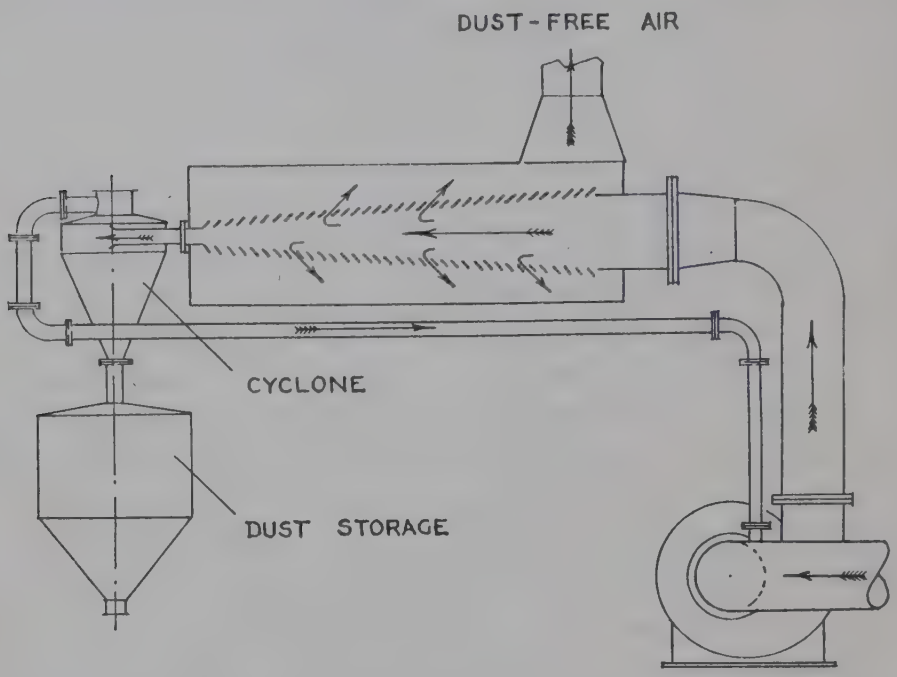


Fig. XII–6.—Musgrave Dust Collector.
(*Courtesy of Musgrave & Co., Belfast*)

there is no doubt that spraying towers would be the most widely used of all air-filtering devices. A plant of this kind is shown in Fig. XII–7; the water contained in the base of the chamber is pumped continuously to the sprays B, which meet the incoming air as it passes through the baffles A. The body of the washer forms a settling chamber, and the washed air leaves through outlet baffles which are designed to remove the last traces of spray. These inlet and outlet baffles resemble a widely set Calder-Fox scrubber and would hardly check much fine mist. The pool of water at the base holds the dust which has been removed and needs changing periodically. The normal air velocity through the spray chamber is about

6 ft. sec., and the pressure drop over the washer is some 8 mm. water gauge.

Spraying towers of various forms have been used instead of the horizontal-flow spray chambers. The essential duty is very similar to that of the same towers used in gas absorption, with the "concentration head" represented by the changing dust concentration. It is probable that the rate of dust removal follows a logarithmic law, and the final stages are very prolonged. No quantitative figures are available for the dust removal, but the towers can be used with gas velocities up to 10 ft./sec., giving a pressure drop of

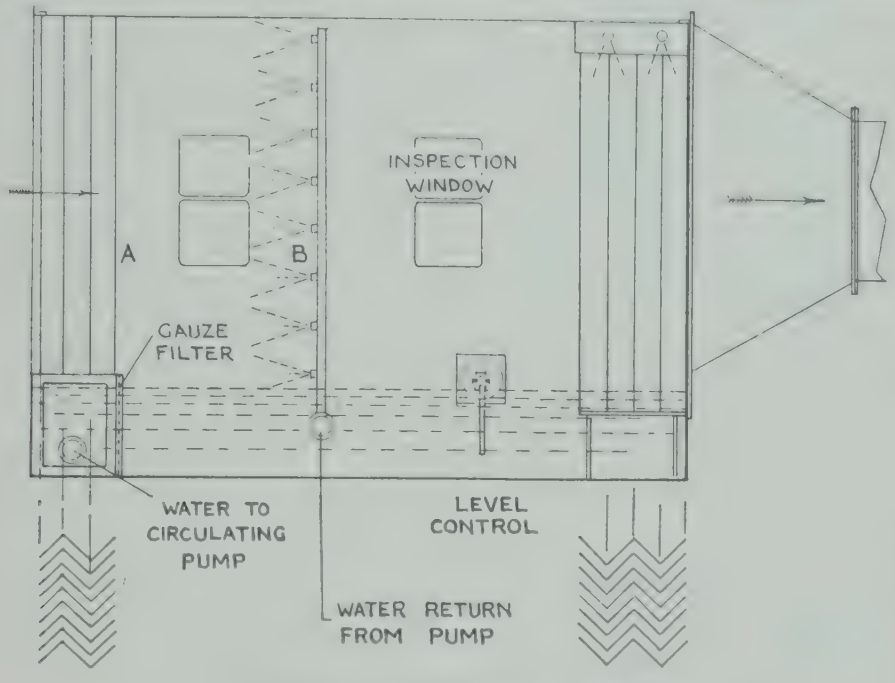


Fig. XII–7.—Air-Washing Chamber.

(*Courtesy of Sturtevant Engineering Co.*)

0·5 in. of water gauge for a 15-ft. height of spray-filled space. If the dust is not easily wetted by water alone, mixtures of oil and water can be used. The Traughber* gas washer, for example, sprays an oil–water mixture into the gas stream and combines this with intensive bubbling through an oil foam. This washer is somewhat reminiscent of the Livesey washers used in the removal of tar fog from coal gas. It may be noted that tar fog used to give much trouble by deposition in the exhausters used to pump the gas from the retorts. It was found possible† to design these exhausters so

* *Ind. Chemist*, 1941, 17, 117.

† Badger, *J.S.C.I.*, 1946, 65, 166.

that they served the dual purpose of pumping gas and precipitating tar fog.

Wet-washing units of the forms described above can only collect the dust as a sludge. Where it is required to keep the solid deposits dry, and at the same time remove finer material than the cyclone can collect, recourse may be made to bag filters. These are of cotton or wool cloth, made up in cylinders about 10 ft. long and 6 in. diameter, and arranged to connect to a common header for the dust-laden gas.

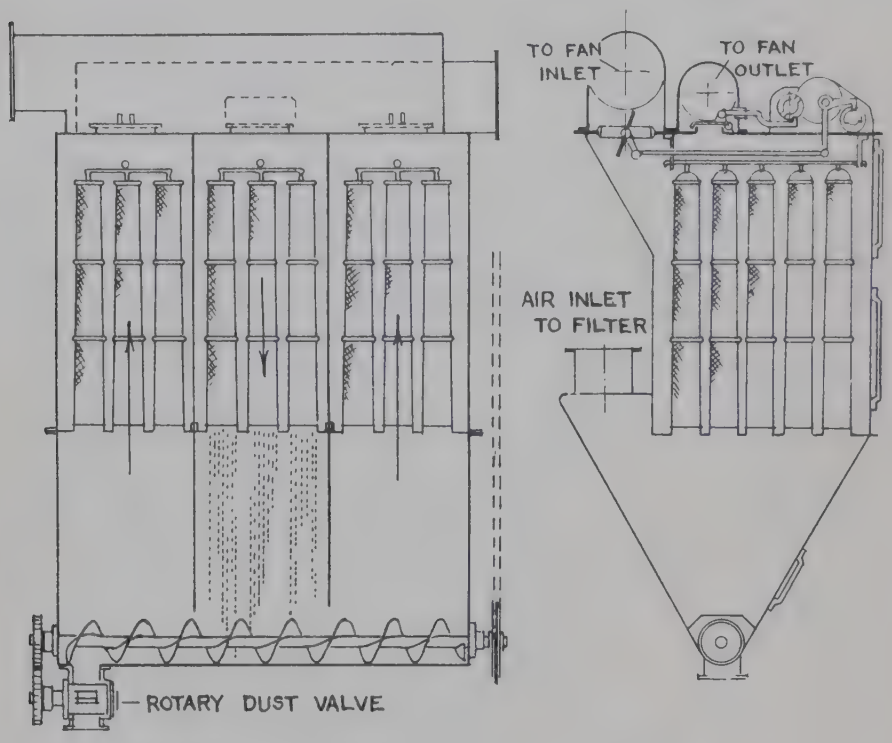


Fig. XII–8.—Bag-Filtering System.

A typical collector will contain 45 of these tube bags arranged in sets of 15, as shown in Fig. XII–8. The air enters at the base of the bags and passes through the fabric to the clean air chamber, leaving the dust behind in the bag. Two sections will be on an air-cleaning cycle, while the third is disconnected by valves and cleaned by an air back-flow. The amount of air which can be filtered in this way is from 1 to 4 cu. ft./sq. ft./min. according to the dust content. The pressure drop is high — up to 2 in. of water gauge — and the fabric limits temperatures to below 100° C. and makes it impossible to scrub gases containing free acid. It has been

suggested* that bag filters should be prepared for filtration by deposition of a filter aid, as in liquid filtration, the filter aid proposed being made from fine flake asbestos.

It is probable that the most efficient of all gas filters is the electrostatic or Lodge-Cottrell precipitator. This form of plant is steadily gaining ground in all fields, despite the high initial cost and the need for high and dangerous electrical voltages. In its simplest form the precipitator consists of a vertical pipe, from 4 to 8 in. diameter, down the centre of which hangs a wire charged to a high negative potential relative to the pipe. The gas to be cleaned passes up or down (usually up) the pipe; the dust particles acquire a negative charge and move rapidly to the outer pipe. There is no question of slower precipitation of the smaller particles, and the speed with which dense mists are removed from a gas stream is remarkable. In a plant treating tar-laden gases from coal carbonisation,† the tar content was reduced from 90 grains to 0·4 grains per 100 cu. ft. by passage through an 11-ft. tube, 6 in. diameter, at a linear velocity of 5 ft./sec., giving a total contact time of just over 2 sec. The central electrode was a star-section wire of $\frac{1}{4}$ in. equivalent diameter, maintained at a potential of 20,000 volts, and current consumption was 8·2 kWh. per million cu. ft. of gas cleaned. The total pressure drop over the entire plant was less than 6 mm. of water gauge.

The efficiency with which a dispersed solid or liquid is removed from a gas phase by this means is proportional to the impressed voltage, and the rate of removal is a function of the dispersed-phase concentration. The voltage drop from electrode to outer wall, which is invariably earthed, is therefore made as great as possible without an arc discharge forming. A 3-in. gap in air has a safe voltage drop of 76,000 volts at ordinary temperatures, but this maximum varies directly with the gas density, and with coal gas of 40% the density of air the maximum voltage is about 30,000. There is always a tendency for the precipitated dust to form a solid structure on the electrodes (a little dust goes to the negative electrode) and this further reduces the allowable voltage. If the deposit is non-conducting, the arcing voltage is lower than in the case of conductors, and it is customary to add moisture or ammonia to the gas before treatment to give a conducting film.

* *Chem. Engng.*, 1947, 54 (No. 8), 113.
† *Gas Journ.*, 1945, 245, 371.

The dust or mist, when precipitated, may run continuously from the bottom of the collecting electrodes. This is the case with sulphuric-acid mists, and the treatment of sulphuric-acid plant effluents is one of the most important applications of this type of precipitator. If an adherent layer is formed, the plant must be shut down periodically for cleaning. A similar arrangement to that described for bag filters, with the plant divided into sections, but with tapping substituted for blow-back, is the usual practice. If the precipitate cannot be dislodged in this way, then it may be possible to add a heavy oil mist to the original gas stream and obtain a fluid sludge on joint precipitation. In a producer-gas plant heavy deposits on the electrodes caused a shut-down for laborious hand-cleaning every two days, but this was completely checked and runs of several weeks obtained by vaporising into the gas stream about 1·5 gal. of a heavy creosote oil per million cu. ft.*

Although the simple tube-and-wire unit is widely used, modern plant has tended to become more complex. When the tube-and-wire plan is retained, the central wire is made from a square section twisted into screw form, while the tubes are made in hexagon section for close packing. These are principally used for mist removal, while dusts are cleaned up in rectangular chambers, with the electrodes as flat plates or curtains of hanging rods. This latter form makes it easy to employ tapping devices to shake off the dust. The gas stream is sometimes passed at a high speed through a very concentrated electrical field—the ionising chamber—followed by a treatment at a slower rate in a large precipitation chamber with a small potential gradient. This reduces the risk of arc formation by the deposits on the electrodes. A number of devices have been used for removal of deposits, but the most thorough treatments rely on moving the electrodes through a scrubbing bath. Thus in one plant the collecting electrodes are mounted on an endless band in slat fashion, and face a fixed ionising chamber for the inlet gas stream. As the slats travel through the gas stream they are charged alternately to opposite polarities. The band descends through the gas chamber and goes into a bath of wash liquid; as the deposits are removed sufficient of this cleansing fluid stays on the slat electrodes to serve as a base for the fresh dust.

The principle of electrostatic dust and mist removal is now very firmly established. Whenever the amount of gas to be cleaned is

* Dougill, *Trans. Inst. Chem. Eng.*, 1945, 23, 1.

sufficient to warrant heavy capital charges, the Lodge-Cottrell plant is almost always installed. The completeness with which all forms of dust can be removed has led to changes in plant design which would not have been possible before the introduction of these precipitators. It is possible to increase rates of reaction and gas velocities far above the level to which they have previously been held by the need for avoiding dust formation. The most corrosive vapours can easily be handled, at temperatures up to the limit of normal plant working, while power consumption is rarely more than 15 kWh. per million cu. ft.

CHAPTER XIII

SIZE REDUCTION

THE main purpose in crushing and grinding solid material is to create additional surface for chemical or physical change. Effective size reduction, therefore, is similar to mixing in that it produces a maximum of surface with a minimum of energy expenditure. It may be that in certain cases the surface formed should possess certain specific characteristics, such as a rigid size range for the constituent particles, but the general rule for efficiency still applies.

Consider a unit weight of any solid material, having a density ρ and a particle size L in linear dimension. Then the weight of each particle will be $K_1\rho L^3$ and the number of particles in unit weight of material will be $1/K_1\rho L^3$. The surface of each particle will be K_2L^2 and the total surface per unit weight will therefore be given by

$$\frac{K_2L^2}{K_1\rho L^3} = K\frac{1}{L}$$

where K_1, K_2, and K $(= K_2/\rho K_1)$ are constants for the material. In order to increase the surface of a solid, it must be ground to a smaller particle size, and it would at first sight appear logical to equate the energy required for size reduction to some function of the fresh surface produced. The general differential equation for an amount of energy dE required to produce a change of dimension dL in particles of a mean size L can be written as

$$dE = -C\frac{dL}{L^n}$$

where C and n are experimental constants. If $n = 2$, then the general equation integrates to

$$E = C\left(\frac{1}{L_2} - \frac{1}{L_1}\right) \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

for a reduction in size from L_1 to L_2. Similarly, when $n = 1$, the same integration gives

$$E = C \log \frac{L_1}{L_2} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

In the first integration the energy required will be proportional to the fresh surface produced, while in the second equal fractional reductions in size require the same amount of energy.

The power requirements for grinding were formulated in these two ways by (1) Rittinger and (2) Kick, but although the original formulae were put forward many years ago, they have never been completely proved or disproved. The bulk of experimental support appeared to be for the surface hypothesis of Rittinger. For instance, Prentice* recorded experiments on the crushing of South African quartzite in which the work required uniformly amounted to 3·5 ft.-lb. per square foot of surface produced. Modern conceptions appear, however, to be moving away from this idea. Thus Poncelet† studied the way in which cubes of solid fractured under direct stress, and came to the conclusion that the first major cracks to form started energy waves which traversed the whole solid mass. They could either develop the surface fissures always present in solids and produce smaller fragments, or be dissipated as heat. The first effect was more likely to occur when the energy wave moved in a small fragment, and the second in the relatively large pieces. The net effect of crushing a cube, therefore, was to produce a number of very small pieces and a few large fragments, while the energy required to start the crushing could only be very approximately connected with the surface produced. This conclusion found support in the measurements of W. F. Carey and E. M. Halton,‡ who found that practically all the energy put into normal grinding systems could be recovered as heat, and no absorption proportional to the surface being produced could be detected.

One outstanding difficulty in the matter lies in the measurement of the surface of irregular solid particles. Even if these granules were of regular form—cubes or spheres—the measurement would be difficult if various sizes were present. Sieving will only separate into size ranges, and the completeness of separation diminishes with particle size. Indirect methods, such as the adsorption of molecules on the surface, rates of sedimentation, and resistance offered to fluid flow by layers of solid particles, all give results which are inconsistent and open to question. It is doubtful whether any exact expression for the surface of a fine powder can be formulated or

* *Bull. Inst. Min. Met.*, 1946, No. 477.
† *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1684, 1944.
‡ *Trans. Inst. Chem. Eng.*, 1946, 24, 102.

determined. In normal grinding practice the feed has a wide size range, while the product may be still more diverse in size. The most convenient form in which to express these varying sizes is in the form of a plot of undersize against reciprocal of linear dimension as in Fig. XIII–1. The area under this curve will be proportional to the surface per unit weight of the material and may afford some indication of the work which must be done to reduce material to this size from a single very large piece. Alternatively, if a feed size and a product size range are plotted on the same diagram, the area formed by these curves and the X axis (Fig. XIII–2) is an indication of the surface formed in the grinding process. If it is assumed, on the basis of Rittinger's hypothesis, that the power required is a function of surface, then areas on the figure become proportional to power requirements.

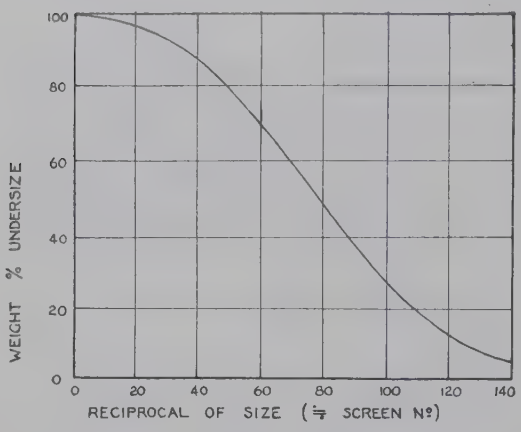


Fig. XIII–1.—Screen Analysis Curve.

Example. *A soft rock, with the size range shown as A in Fig. XIII–2, required 5 h.p. hr./ton to crush to the product B. Estimate the power required to crush this rock to a product of the characteristics shown at C.*

The area between A, B, and the X axis is 50 units, and that between A, C, and the X axis is 100 units. The power required to pass from A to C will therefore be 5 × 100/50 or 10 h.p. hr./ton.

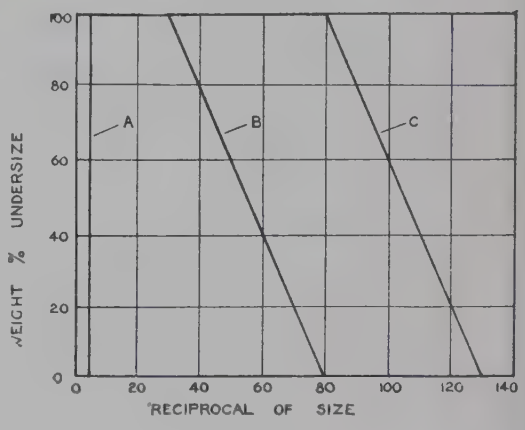


Fig. XIII–2.

In actual practice such sharply defined curves as those shown in Fig. XIII–2 are not obtained. The very long "tail" shown in Fig. XIII–1 is usual. This may represent a large proportion of the total surface, and several attempts have been made to determine the

mathematical form of this " tail ". It has been suggested* that a straight line is obtained by plotting, with logarithmic co-ordinates, the weights retained by a series of sieves of regular size range against the mean particle size of each fraction. The simplest mathematical expression for this relationship is $W_n = C \times L/a^n$, where W_n is the weight of a fraction of linear dimension L/a^n in the series L/a, L/a^2, L/a^3 . . ., and C is constant. As the surface per unit weight of material has already been shown to be equal to K/L the surface of each size fraction will be $(C \times L/a^n) \times K/L \times a^n$, or $C \times K$. As this is independent of L it follows that the solid surface of each fraction is constant. Again correlating surface and work, this shows that the same amount of work is required to produce 1 lb. of material of 1 in. size as to produce 0·25 lb. of $\frac{1}{4}$ in. or 0·0625 lb. of $\frac{1}{16}$ in., and so on. Although in this simple form the rule is far from correct, it

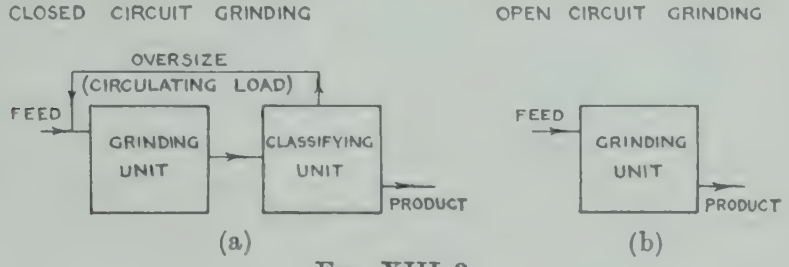


FIG. XIII-3.

illustrates the large amount of work which may be required to produce comparatively small amounts of very fine material. A lower limit to this extrapolation of values for size distribution has been set by the assumption† that no particles of less than 0·001 mm. can be produced by grinding, but in view of the grave doubts which exist as to the whole relation between power requirements and surface the matter will not be taken further here.‡

Whatever the form of the size curve, or whatever the reliability of basic equations, it is experimentally true that the production of a large amount of unwanted fines is very wasteful of power. For this reason all fine-grinding and many coarse-grinding or crushing plants are provided with some type of classifier and grind in " closed circuit ", as in Fig. XIII-3a. The open-circuit grinding shown in Fig. XIII-3b will produce a very wide spread of particle size in the

* Gaudin, *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1779, 1944.
† Warnecke, *Chem. Eng. Min. Rev.*, 1946, 38, 323.
‡ For a discussion on this see Taggart, " Ore Dressing ", 1946.

product. This may be desirable in certain industries, but where a size specification calls for all material to pass through a certain size of screen, with no further definition, then open-circuit grinding becomes very uneconomic. In the case of coarse grinding or crushing, the classifying plant may be dispensed with by crushing in stages, with a " reduction ratio " of about 4 to 1 in linear dimension at each stage.

Crushing. The largest solid fragments which are met with in crushing practice are of the order of 12 in. in size. These are the biggest pieces which can conveniently be removed from mines or quarries, though some quarrying operations do produce fragments up to 24 in. in size. As a rule such fragments are broken down at the quarry face by hand. In crushing, these solids are reduced to about $\frac{1}{2}$ to $\frac{1}{4}$ in. in size before passing on to the grinding plants. The best size for changing over from crushing to grinding plant is determined by the nature of the material being handled. Thus Farrant* suggests for a normal hard rock an economic size of $\frac{1}{4}$ in., as shown in Fig. XIII–4. Very soft solids, such as dried filter-cake, may be fed to grinding units in 1-in. pieces, while a very hard abrasive rock should be crushed to $\frac{1}{8}$ in. before changing over.

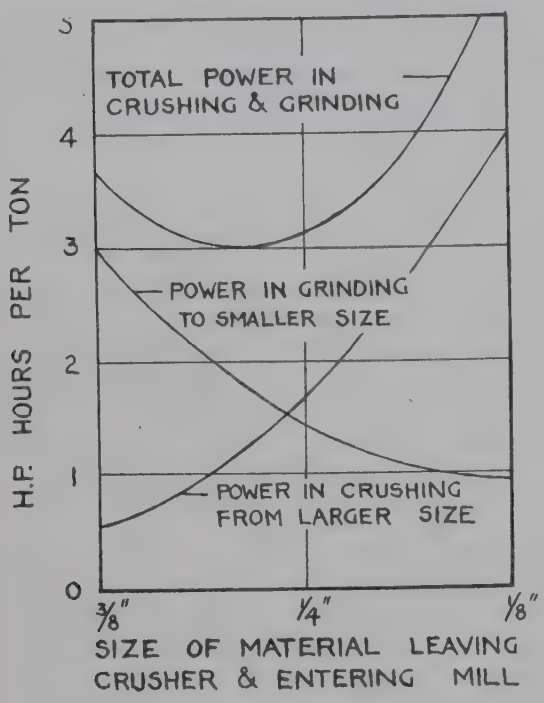


Fig. XIII–4.

The most typical plants for crushing are swing-jaw or gyratory crushers, with rolls as a final stage. The swing-jaw crusher, the most widely used primary crusher for all loads up to 10 tons/hr., is usually built in the form shown in Fig. XIII–5. A moving jaw is pivoted at the top and moved to and fro against a fixed crushing surface by a heavy eccentric bearing. The maximum movement of the jaw is at the bottom or discharge opening, and there is only actual crushing at the forward stroke of the jaw. It follows from this

* *Trans. Inst. Chem. Eng.*, 1940, 18, 56.

that the discharge will tend to be uneven in size, and that the load on the driving unit will be uneven, although cushioned by a heavy flywheel. The jaw crusher is therefore noisy and causes a considerable amount of vibration. A more even discharge may be secured by pivoting the moving jaw at the bottom, but this design was found to lead to choking and is rarely used, except for the finer stages of crushing. A rearrangement of the jaw to give a rolling action instead of a straight to-and-fro motion has proved more successful, both in giving a more even size in discharge and in reducing vibration. This "roller jaw" crusher is shown in Fig. XIII–6, and it will be seen from the diagram that the crushing is spread over a much longer portion of the jaw channel. The small connecting links transmitting motion to the jaw from the eccentric are known as toggle bars, and are split and jointed with soft-iron rivets. If any material drops into the crusher which is too hard to fracture and too large for the loaded springs to pass, the toggle rivets shear and little or no harm is done to the machine. As broken iron tools are often met with in crushing, this precaution is very necessary.

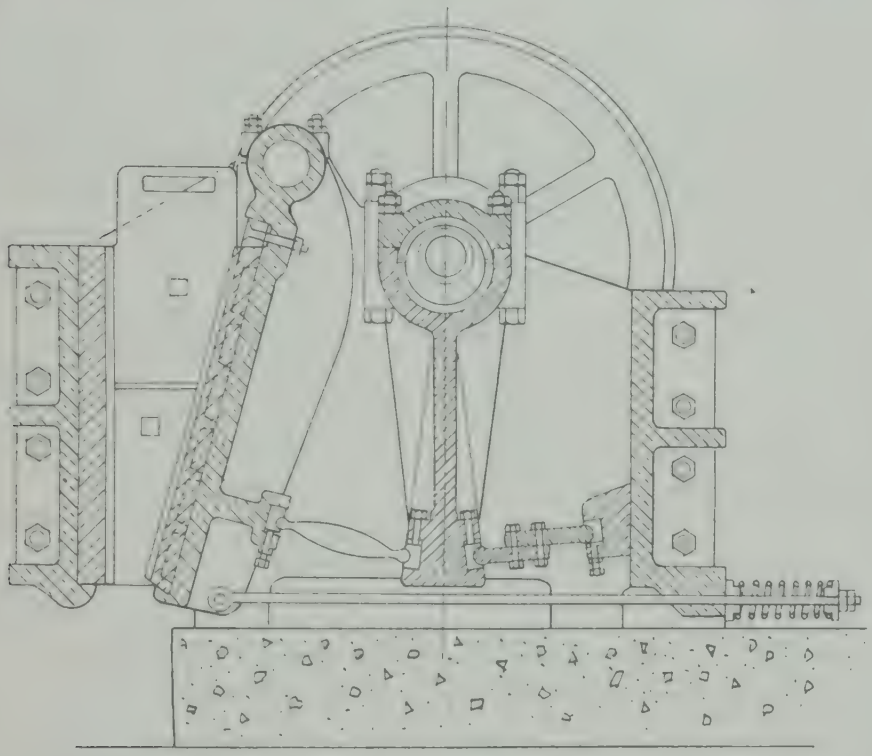


FIG. XIII–5.—JAW CRUSHER.

The ratio of jaw-opening width to jaw-discharge width is not more

than 6 to 1 and usually somewhat less. No solid with all dimensions greater than the maximum width of the discharge opening can escape from the crusher, so classification and recirculation of oversize is not necessary. Of course, no jaw crusher set to a specific opening will give a product of a single size. A wide spread of particles below the dimension of the opening and a few of slightly above this size form the characteristic material. The discharge opening should be set to some 15% above the required particle size, as this appears to give the greatest output of suitable material.

Consideration of the action of a jaw crusher shows that the crushed product is in "free" fall for the whole time that the jaw is swinging back from the fixed plate. Ideally, all the material broken to size by the forward stroke will drop out on the back stroke. If this material follows the general rules for flow of granular solids, the rate of fall will not be affected by the layers of material above the discharge opening, and a strip of depth proportional to $\frac{1}{2}gt^2$ will drop in a time t. If this time t is so adjusted that a strip of depth d, equal to the width of the discharge opening, falls clear at each stroke of the jaw, then $d = \frac{1}{2}gt^2$, or $t = \sqrt{(d/193)}$ (d in inches). The value of t so obtained may be called the natural period of the crusher, giving a speed of $417/\sqrt{d}$ r.p.m. Actual working speeds are generally about 60% of this rate. This could be expected from the various hindrances to free fall which exist at the outlet opening. When the jaw is working at 60% of the "natural" speed, an opening d in. wide and l in. long may be assumed to give a discharge of ld^2 cu. in. per stroke, or $0{\cdot}6 \times 417ld^{1{\cdot}5}$ cu. in./min. Assuming a crushed rock density of 0·05 lb./cu. in., the output will be $12{\cdot}5ld^{1{\cdot}5}$ lb./min., or $0{\cdot}34ld^{1{\cdot}5}$ tons/hr. This appears to be in reasonable agreement with working figures. Several other formulae for jaw crushers are available, but this seems to be the most rational.

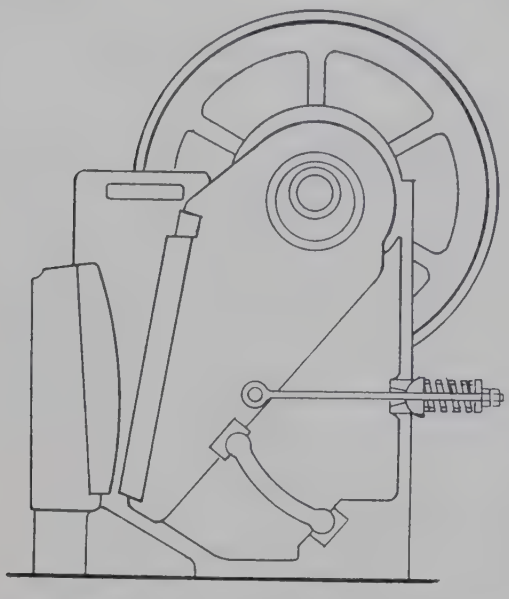


Fig. XIII-6.—Roller Jaw Crusher.

The power requirements for crushing plant may be expressed in the form of "reduction tons per horse-power hour", where the "reduction ton" is the product of output weight and reduction ratio. The latter has been further defined as the ratio of the square mesh openings which will pass 80% of the feed and product respectively. The figures for crushing will vary widely with the width of discharge opening, a point which is manifested in the table below.

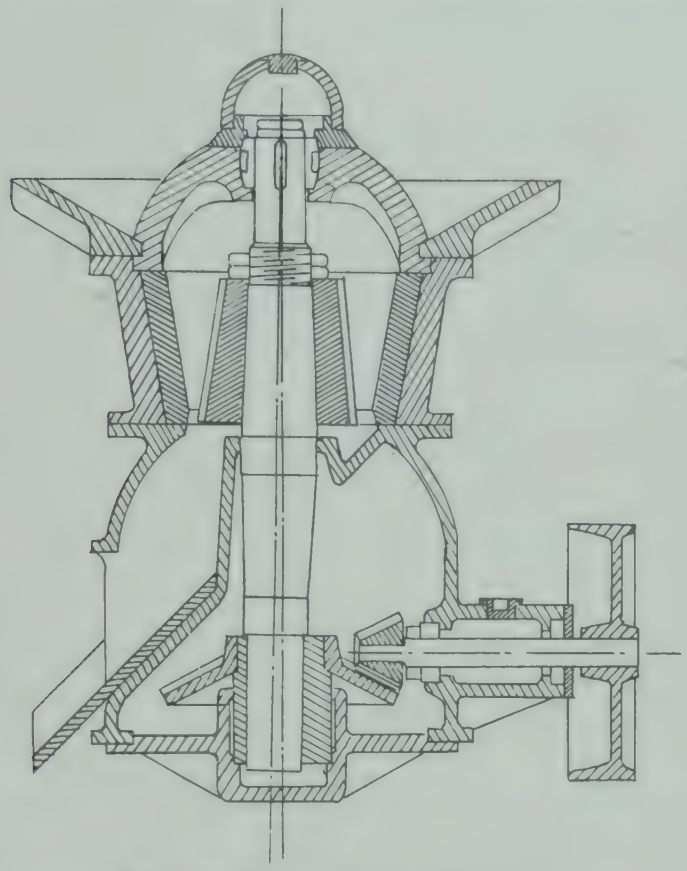


FIG. XIII–7.—GYRATORY CRUSHER.

This has been built up from collected catalogue figures for a medium rock (e.g. dense limestone) with a reduction ratio of 4–6.

Discharge Opening . . .	1 in.	2 in.	3 in.	4 in.	5 in.	6 in.	10 in.
Reduction tons per h.p. hr. .	2	3	4	5	8	10	12

In the crushing of hard granite these outputs will be reduced by 25–30%, but a light brittle material such as coal shows relatively no change from limestone in the output per h.p. hr. This may be due to a less even flow through the crusher.

The alternative to the jaw crusher, especially for large loads, is the gyratory crusher, as shown in Fig. XIII–7. The inner crushing

"mantle" does not rotate inside the fixed breaking surface, but gyrates under the influence of the eccentric bearing at the foot of the crusher. The feed enters at the top of the annular space between the two ridged crushing surfaces and flows down through a steadily decreasing path area, passing out finally through a zone of almost constant cross-section. This final stage is a recent development, and has allowed the gyratory crusher to be used in the production of a rather finer product than had been the case with earlier forms. The gyratory motion means that a crushing "wave" is passing round the annulus, and ideally a ribbon of crushed material is being unwound at the discharge opening. As crushing at some point is always going on, the load is much more even and there is less vibra-

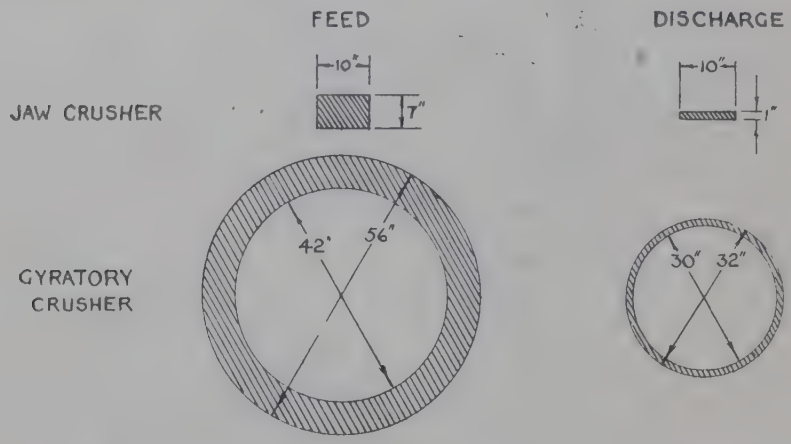


FIG. XIII–8.—CRUSHER FEED AND DISCHARGE OPENINGS.

tion than in the case of a jaw crusher. It would appear, however, that maintenance costs are higher than in the case of the jaw crusher, and it is unusual to find a gyratory crusher being used for any load of less than about 100 tons/hr. One reason for this is that the circular form of feed and discharge opening limits the minimum size of this type of crusher for any particular size of feed and reduction ratio. In the jaw crusher, on the other hand, the length l of the feed and discharge opening can be reduced to almost the width of the feed opening. This point is brought out in Fig. XIII–8, which shows the relative areas of feed and discharge for typical jaw and gyratory crushers set to produce 1-in. fragments from a 6-in. feed.

Where a gyratory crusher can be used, it is more economical in power, giving up to three times the reduction tons per h.p. hr.

obtained with a jaw crusher, with a very similar increase as discharge size increases. Once again a rational speed would be given by the rate $417/\sqrt{d}$ r.p.m., but this is definitely higher than the practical working figures, which seem to indicate about 50% of this speed. Then the throughput, based on a discharge opening of area A and width d, will be about $(Ad) \times 417/2\sqrt{d} \times 0{\cdot}049$ lb./min., or $0{\cdot}27A\sqrt{d}$ tons/hr. Results from this formula appear to agree reasonably well with published figures.

A possible alternative to the two types of crusher described above is the toothed or "sledging" roller crusher, which is sometimes used to break up brittle material such as coal, clay, or soft phosphate rock. The output and power requirements appear to follow the general rules of roller crushing, as described below. Some forms of hammer mills can also take feed sizes up to 12 in., and may be classed as crushers, but are best dealt with in the chapter on grinding. These primary crushers will reduce the size of a feed, in one or more stages, to about 1 in. with ease, and may be used to give smaller fragments, but size reduction from 1 in. to $\frac{1}{4}$ in. is most conveniently carried out by rollers.

Roller mills are built up from two rolls, rotating towards each other at the top, and mounted in a frame with one roller free to move back against the action of heavy springs. The rollers are driven separately from a suitable power source, and are generally smooth when first fitted, though corrugations soon appear. The shell of the roll may be of specially hardened steel, mounted on a mild-steel core, so that the shell can be changed when too badly worn. The speed of these rolls varies from 1000 ft./min. (peripheral velocity) up to 3000 ft./min. for the very largest rolls of 6 ft. or more diameter. The feed passes into the cavity at the top of the rolls and is dragged down by the combined force of gravity and friction, as shown in Fig. XIII–9.

This places definite limitations on the size of feed. Considering the diagram of Fig. XIII–10, a feed particle of diameter d rests between two rolls of diameter D with clearance S and is subject to a tangential force acting downwards, resisted by a normal reaction outwards. The angle of nip of a roller crushing system is defined as the angle made by the tangents to the faces of the rolls at the points of contact of the particle being crushed (A, B, C in the figure). Then the feed fragment of Fig. XIII–10 will just "ride" the rolls when the vertical component of the normal force, $N \sin \alpha$,

is equal to the vertical component of the tangential force, $T \cos \alpha$, where α is half the angle of nip.

Then $T \cos \alpha = N \sin \alpha$, or $T/N = \tan \alpha$. This is the normal formula for the angle of slip of a particle on an inclined plane.

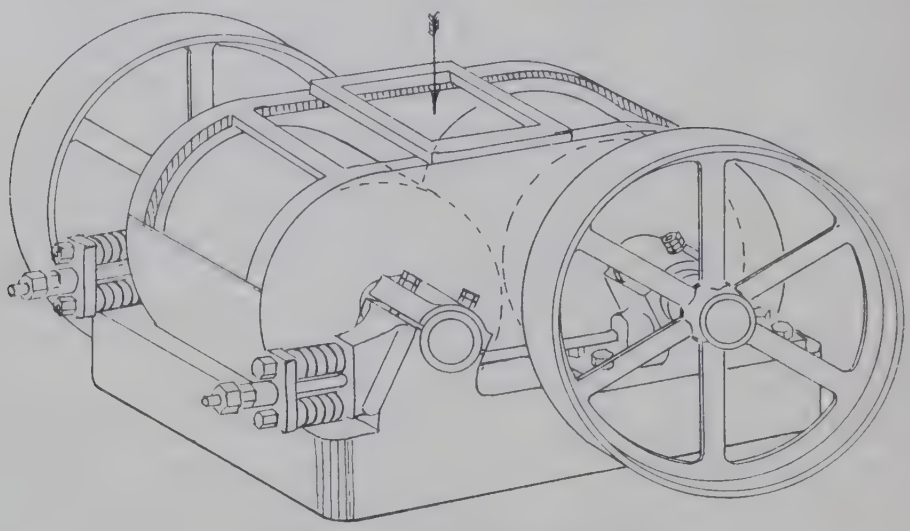


Fig. XIII–9.—Crushing Rolls.

For stone on iron, $\tan \alpha$ is approximately 0·3, whence $\alpha = 16° 42'$ and the angle of nip is 33° 24′. It can also be seen from the figure that

$$\cos \alpha = \frac{D + S}{D + d} = \cos 16° 42'$$

In practice the nip angle rarely exceeds 25°, and may be as low as 5°.

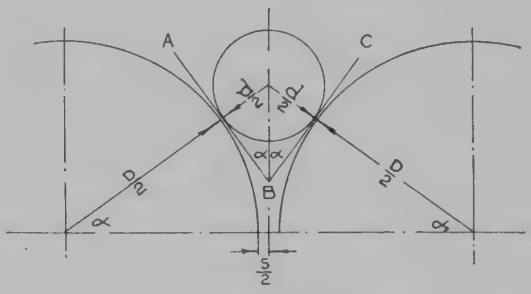


Fig. XIII–10.—Roller Crusher Diagram.

The mean value for a wide range of values quoted by Taggart* is 13°. For the maximum value of 25°

$$\frac{D + S}{D + d} = \cos 12° 30' = 0{\cdot}9763, \text{ or } d = S + \frac{D}{41}$$

$$\text{Approximately, } d = D/40$$

* Taggart, " Ore Dressing ".

A ribbon of crushed material will be discharged from the rolls, at a rate equal to the peripheral speed, and with a thickness approximating to S, the "set" of the machine. The throughput cannot be based very exactly on this ribbon weight, as the feed forces the rolls apart to an indeterminate extent, especially with settings of below $\frac{1}{8}$ in. For such settings the output is about three times the weight that would be expected on a ribbon formula based on the packing density of crushed rock. The power requirements for these rollers are not greatly affected by the kind of material being crushed, but are determined by the methods of feeding and the set of the rolls compared to the diameter. If these are so arranged that the rolls are always forced apart against the spring pressures—"choke feeding"—it is possible to obtain as much as 10 reduction tons per h.p. hr., with reduction ratios of 3–6. For "free" crushing, with a slower rate of feed and no motion of the rolls, the output may drop to 1–2 reduction tons per h.p. hr., but an even-sized product is obtained, with very little oversize or fines.

In addition to roller crushers, special forms of gyratory crusher have been designed to operate in the size range 1 in. down to $\frac{1}{8}$ in. These have a higher speed and eccentricity than the plant for larger sizes, with cones of a more obtuse angle and a longer zone of even area. Another alternative is a double-cone unit of similar section to the gyratory plant, but with a rotating movement of the inner cone. Corrugations on this cone are designed to draw the feed down into the crushing space. Hammer mills have been used again in this field, as with coarse crushing, but neither these nor the special forms above appear to have any notable advantage over roller mills.

Grinding. The process of size reduction from $\frac{1}{4}$ in. down to the finest particles used in the chemical industry is carried out by three general forms of grinding plant. These may be briefly classified as follows:

(1) Low-speed unit—low maintenance; grind by attrition and impact.
(2) Medium-speed unit—low power; grind by attrition.
(3) High-speed unit—low cost; grind by impact.

Low-speed mills are rotating cases, in which the grinding surfaces (balls, pebbles, or rods) roll and tumble over each other while the material being ground fills the interstices. The process can be very easily adapted to either wet or dry grinding.

In wet grinding, the feed is suspended in water, slurries of 30–60% solids being used. Although there appears to be little difference in the power consumed in the actual grinding, a classification plant using water is less costly in power than one which uses air or even vibrating screens. This point will be taken up later, in Chapter XIV, but provides the principal reason why low-speed mills are operated for wet grinding whenever possible. As medium- and high-speed grinding plants are not well adapted to wet processing, slow-speed units are the most widely used of the three classified above.

The simple ball mill, as shown in Fig. XIII–11, is a cylindrical steel shell, lined with an abrasion-resisting material and holding a charge of hardened metal balls. The diameter of such a mill varies from 3 to 10 ft., with a length usually slightly less than the diameter.

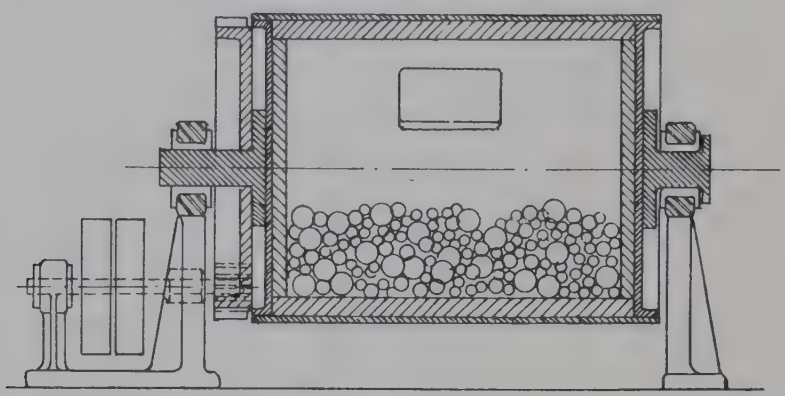


Fig. XIII–11.—Ball Mill.

The ball charge is sufficient to fill about 40% of the total mill volume, and is proportional to the size of the feed. If the individual balls are considered as small roller crushers, then a $\frac{1}{8}$-in. feed would need a 5-in. diameter ball. Alternatively, it has been suggested that the ball should be of such a size as to break the largest feed particle when dropped a distance equal to the mill diameter. The biggest ball used in practice is 6 in. diameter, and it may be necessary to crush very hard feeds to below $\frac{1}{8}$ in., as suggested above.

The grinding balls are now always spherical, though other forms have been tried in the past. In any case, an irregular form wears to a sphere fairly quickly, though badly worn metal balls sometimes assume a dumb-bell form before breaking at the " neck ". Chilled cast iron is the most common metal for fabrication, but forged chrome steel has been recommended. An economic balance has to be struck between first cost, depreciation, and the effects of metal

contamination of the material being ground. In typical grinding work the time taken for a 3-in. ball to wear down to 1 in. varies from 60 to 150 days according to composition and method of fabrication.* This corresponds to a consumption of 1–3 lb. of metal per ton of material passing through the mill. The whole question of ball wear is very complicated and bound up in the corrosive qualities of the slurry, as well as in the abrasive qualities of the solid being ground.† It has been maintained‡ that the rate of wear is proportional to the ball surface. That is to say, if $\frac{1}{8}$ in. of metal is worn from a 3-in. ball, $\frac{1}{8}$ in. will also be worn from a 1-in. ball. It follows that the bulk of size reduction in a ball mill must be by attrition, a view which is not supported by all the evidence available.

The lining of a ball mill is also very important and a number of materials have been tried out, including rubber. This last reduces wear very considerably, but cushions the ball action to such an extent that the output drops to an uneconomic value.§ Hard metal linings made in the form of steps, with each step so dimensioned as to trap a ball of the grinding charge, will last for twice as long as a smooth lining made from the same materials, while grinding efficiency is slightly improved.‖

The feed into the mill can be by a spiral scoop, dipping into a slurry tank, as shown in Fig. XIII–12a, or by a hopper discharging into a trunnion conveyor (Fig. XIII–12b). These are the methods usually adopted for wet and dry working respectively. The discharge is usually by an open trunnion overflow—the so-called "high discharge". Alternatively, the ball charge may be restrained by a perforated grate, and lifters in the space beyond raise the pulp to the discharge outlet—"low discharge". The mill of Fig. XIII–12b is fitted for the former, and that of Fig. XIII–12a for the latter. As a large ball mill and its load may weigh more than 20 tons, the drive for these large rotating loads presents some mechanical difficulty. Carefully cut spur gearing, with a large gear-wheel belt running round the mill, is the most general method, but a central drive through a long connecting-rod fixed to the mill end has been claimed to effect considerable economies in power.¶

* *J. Chem. Met. Soc. S. Africa*, 1943, 43, 99.
† *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1918, 1945.
‡ *J. Chem. Met. Soc. S. Africa*, 1944, 44, 179.
§ Banks, *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1890, 1945.
‖ Howes, *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1577, 1943.
¶ Edgar Allen & Co., Ltd. (manufacturer's claim).

The bearings, whatever their type, must be carefully made despite the slow speed of rotation, and roller bearings have been used. The bearings of a large mill should be water-cooled.

When the ball mill is rotated, the ball charge is set in motion, with the balls rolling from the upper side of the grinding mass to the lower. If the speed of rotation is steadily increased, individual balls will tend to leave the mass and fall freely to the bottom of the mill ("cascading"). As the speed is increased still further, the ball mass will be held against the mill casing by centrifugal force and grinding will cease. At the point where motion ceases, the weight

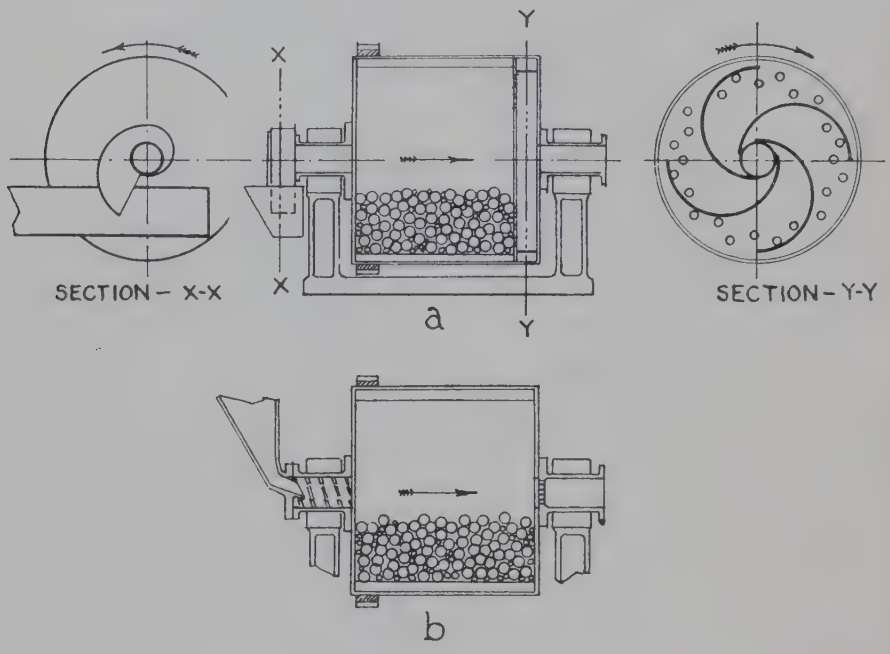


FIG. XIII–12.—BALL-MILL FEED AND DISCHARGE.

of the ball acting downwards balances the centrifugal force acting outwards. For a ball mill of effective diameter D ft., rotating at N revolutions/sec.

$$Mg = \frac{2(\pi DN)^2 M}{D}, \text{ or } N = \sqrt{\frac{1{\cdot}62}{D}}$$

If the speed is given in r.p.m., this formula reduces to $76/\sqrt{D}$. The effective diameter is equal to the mill-shell diameter less two ball diameters. Normal working speeds are from 60–90% of the theoretical "critical speed", and over this range the output of a mill will increase with the speed, but the power requirements will increase at least as rapidly. The curves of Fig. XIII–13 illustrate

this point. As there is little or no gain in output per h.p. hr., the greater wear on bearings, together with an increase in ball breakage, sets a practical limit at about 75% of the critical speed. Higher ranges of speed are chosen for reduction to 40-mesh material and lower ranges for finer grinding.

The rate at which the material is fed to the mill, if other conditions are maintained constant, will determine the degree of size reduction and the power required for the production of a definite size range, as shown in the following table.

Feed Rate (lb./hr.)	% below 65-mesh in Discharge	H.p. hr./ton of 65-mesh
1000	60	17·8
2000	48·5	11·0
3000	40·0	8·9
4000	35·0	7·6
5000	33·0	6·5

All figures for a 3-ft. diam. mill, working on $\frac{3}{8}$-in. limestone.

Clearly for high outputs of any specific size range it is necessary to install a classifier and to return a certain proportion of the product as a " circulating load ". The power required for the classifier will, of course, diminish the apparent economy, but is usually well below the power required for the mill. For the operation covered by the table above, a very small classifier, taking less than 1 h.p., would deal with all the solid being handled. There are no theoretical limits to the increase in output to be obtained from a grinding unit, but in practice the accumulation of medium-sized particles would very soon choke both classifier and mill if circulating loads of more than six times the feed or product rates were attempted.

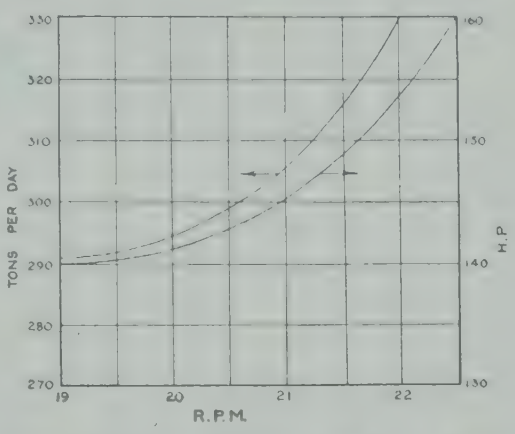


Fig. XIII-13.—Output of Ball Mill.

The ideal ball-milling process would use large balls to crush the largest feed particles and then smaller balls to complete the grinding. This may be achieved by making the mill much longer than usual—the so-called " tube mill "—and dividing the mill length by screens.

The screens are difficult to maintain in an effective condition, and the tube mill has been largely abandoned in favour of the pear-shaped Hardinge mill shown in Fig. XIII–14. The shape of this mill tends to concentrate the larger balls at the end nearest the feed, while the smaller pass down the tapering cone of the discharge end. While this segregation is desirable, the mill cannot be driven at its most effective speed for all the varying diameters of grinding space. It has, however, been very widely used.

It is unusual to find a mill with internal screens to separate the ground products from the rest of the charge, though this principle was employed in some of the first ball mills used in the chemical

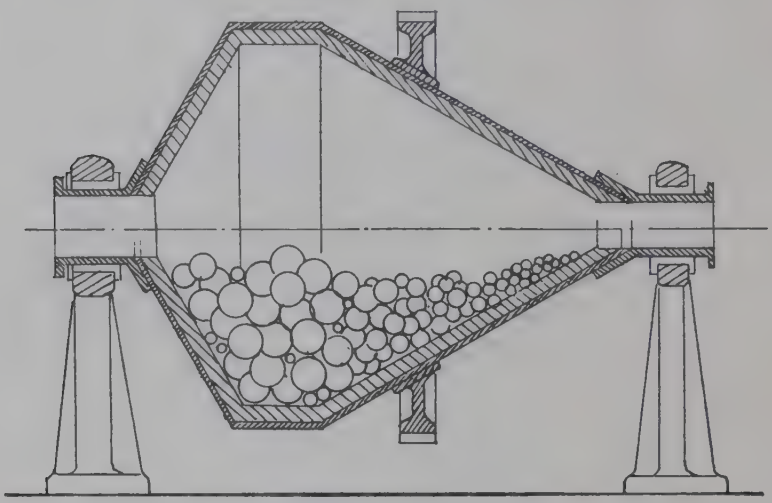


Fig. XIII–14.—Hardinge Ball Mill.

(*Courtesy of International Combustion, Ltd.*)

industry. A modern mill of this type is shown in Fig. XIII–15, and, as will be seen, discharges fine material continuously through the screen which forms the inner lining of the grinding chamber. It is recommended by the makers for dry grinding, and a mill of 6 ft. diam. by 3 ft. 6 in. long is driven at the high speed of 27 r.p.m. The speed is higher than usual, but the effective grinding may be done on a much smaller diameter than 6 ft.

The grinding in a ball mill may be on either a slurry of solid and water containing up to 70% of solids, or on a dry solid with less than 2% of water. When the solid is being ground "dry" the water content should be as low as possible, or the mill output drops sharply. Power consumption, or mill throughput when driven by a fixed power, is about the same in either case, but the wet classifiers used

are more efficient than air classifiers. Screens are now being substituted for classifiers to some extent and may swing the balance to dry working, while ball and liner wear is much less in dry than in wet mills. As a rule the choice between wet and dry grinding must be based on the requirements of processing before and after grinding.

The power requirements for grinding vary much more widely than those for crushing. The effect of the material has been referred to as a " grindability rating ", but no standard method has been devised for measuring this property, and it is extremely doubtful if any one method, holding for all sizes or reduction ratios, will

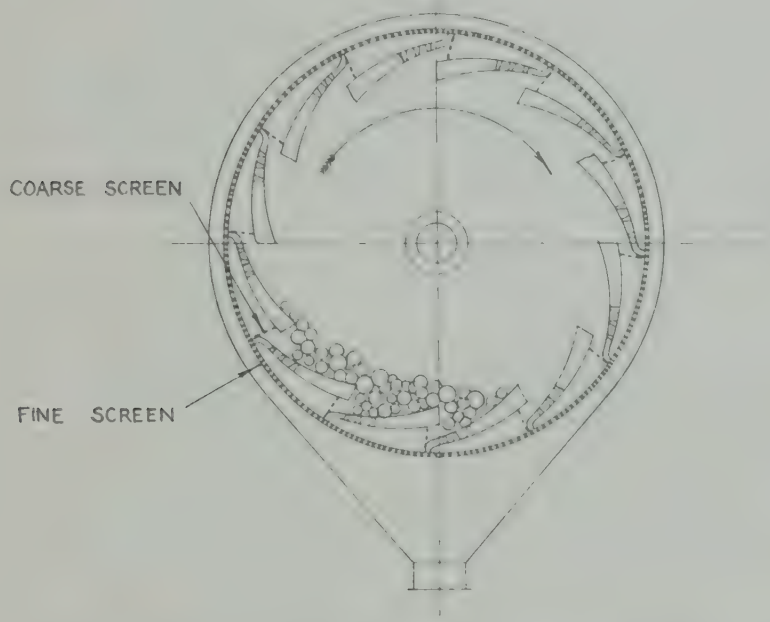


FIG. XIII–15.—BALL MILL WITH INTERNAL SCREEN.
(*Courtesy of Edgar Allen & Co., Ltd.*)

ever be devised. Simple comparative processes have been suggested, but it is perhaps better to match the case being studied to one of the numerous processes for which numerical results are available in literature. Farrant,* Taggart,† and Perry‡ all give a number of such processes. The figures are very difficult to reduce to a standard form, such as reduction tons per h.p. hr., as a detailed screen analysis of feed and product is seldom supplied. For a feed of $\frac{1}{4}$-in. size and medium hardness, the output per h.p. hr. is 0·15 tons

* *Trans. Chem. Eng. Grp.*, 1931, 13, 9.
† " Ore Dressing ".
‡ *Chem. Eng. Handbook.*

in reducing to 40-mesh, and 0·07 tons in reducing to 100-mesh (British Standard Sieves), while the power required to drive a mill varies from 0·5 to 1 h.p./cu. ft. of grinding volume.*

As an alternative to steel or iron balls, flint stone pebbles may be used, and have the advantage that metallic contamination is reduced to a minimum. They can be combined with hard porcelain linings, and these can be built up from sections to line any size of mill. Balls made from heavily fired earthenware can also be used, but are rather light for hard rocks.

Rod mills, in which the grinding surfaces are steel rods, are claimed to use less power than ball mills and to produce a more even product.

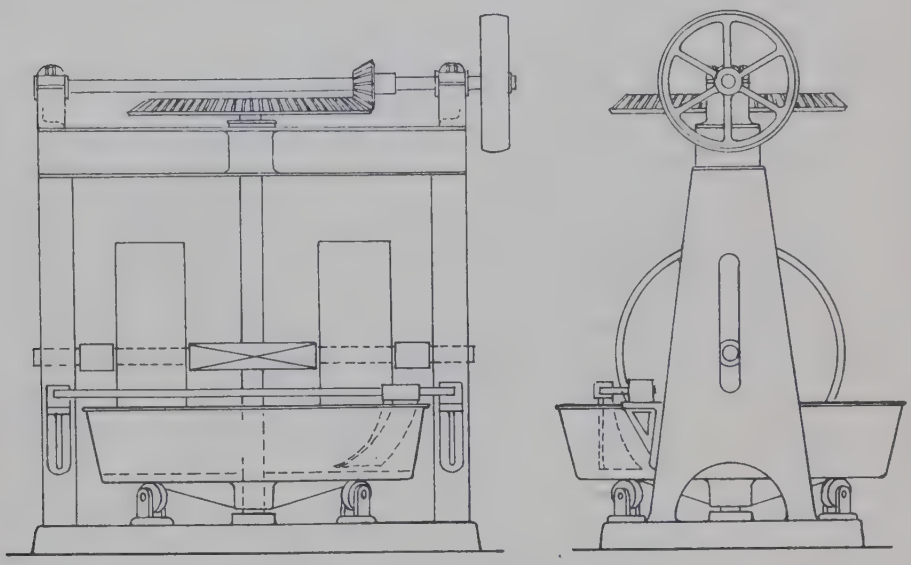


FIG. XIII–16.—PAN MILL.

Breaking and distortion effects do not seem to be in any way serious, and it may be that the use of rods will extend in the future.

Medium-speed units for grinding have probably developed from the pan mill shown in Fig. XIII–16. Mills of this kind were probably the earliest of all grinding appliances, and have been in use, almost in the form shown, for hundreds or perhaps thousands of years. The author has seen such a mill, used for grinding agricultural limestone, which had been in service for over 200 years, with horses as the motivating power. The wheels were of stone, and of sufficient weight to crush the lumps of feed. As mechanical mills were introduced and speeds increased, the action became one of

* See, e.g., Brown *et al.*, *Inst. Fuel Conf. Pulv. Fuel*, 1947.

attrition, and the modern mill relies on this to a very great extent. The weight of the original rollers has been replaced by spring or centrifugal forces, but the rollers still rotate inside a fixed casing. In the Sturtevant mill, as shown in Fig. XIII–17, three rollers are pressed by springs against a crushing ring, which forms the moving member. It rotates at speeds up to 200 r.p.m., and by frictional forces sets the rollers rotating on their axes. The springs holding the rollers against the ring exert a force of from 10 to 30 tons, and the material to be ground is flung into the space between the ring

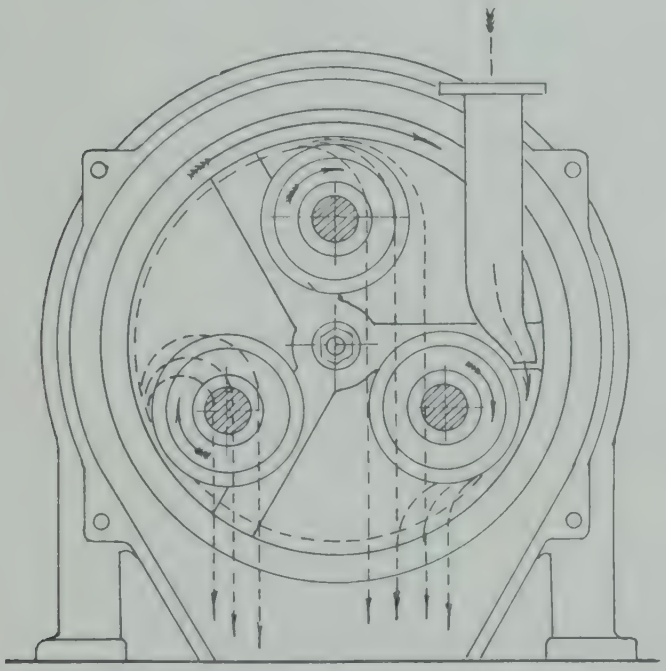


Fig. XIII–17.—Sturtevant Ring Roll Mill.
(*Courtesy of Sturtevant Engineering Co.*)

and the roller by centrifugal drag, forcing the rollers away from the ring. The mill is not often used for very fine grinding to below 100-mesh, and outputs vary to a considerable extent. A 40-in. diam. roll, 8 in. wide, with three rollers, gave an output of 7 tons/hr. in reducing a soft phosphate rock from $\frac{1}{2}$-in. feed to a product of 85% through a 60-mesh screen. A 75 h.p. motor was used for driving, but it was suggested that the mill was too highly powered.

The Sturtevant mill is typical of several other units in which the grinding surfaces are held together by springs, which tend to weaken with continued use and must be replaced periodically. An alternative is to use the centrifugal force available at speeds of

150 to 200 r.p.m. In the Raymond mill shown in Fig. XIII–18, the rollers are mounted on the ends of top-pivoted arms which are free to swing out as in a mechanical governor. In rotation, then, they press against the grinding ring, which is the fixed member in this form of mill. Ploughs mounted on the same central axis as the rollers fling the material to be crushed up into the grinding zone. The mill is air-swept, with a classifying unit built on the top, and all fines are carried away, along with the medium-sized fragments, as

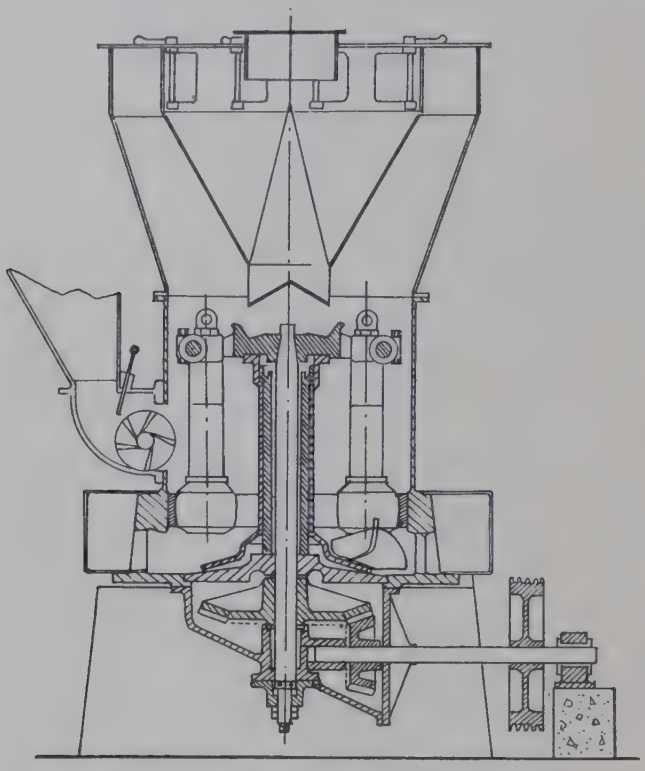


FIG. XIII–18.—RAYMOND MILL.

(Courtesy of International Combustion, Ltd.)

soon as they are formed. In the Sturtevant a steady flow of material through the mill removes fines, oversize, and uncrushed feed as a heterogeneous mixture. This air-sweeping is not an unmixed improvement, as the power required to force air through mill and classifier represents a large proportion of the total used for grinding. Thus a mill grinding hard coal to 200-mesh at a rate of 2 tons/hr. required 40 h.p. for grinding and an additional 30 h.p. for the fan. A further discussion on this matter is included in the Chapter XIV. The Raymond mill construction is complicated and therefore costly. A cheaper form of mill is the Griffin mill of Fig.

XIII–19. Here a single roll is kept against the ring by centrifugal force, while a scraper picks up the feed from the base of the unit and throws it against the grinding track and the screen above. The fines are removed from the oversize, which drops back for regrinding. A mill with a 30-in. ring and an 18-in. roll ground 2 tons/hr. of limestone from $\frac{1}{2}$-in. feed to 80% under 200-mesh, while taking 30 h.p. for driving. For a similar load,* a ball mill took 30 h.p. per ton, or twice as much power, while a Raymond mill of the type above required 20 h.p./ton. It must be remembered, in comparing these figures, that an exact comparison between one mill and another cannot be made on such simple data. The shape of the final particle and the form of the screen analysis curve are determined by the characteristics of the grinding machinery used, and these factors may or may not facilitate subsequent chemical operations at a later stage in the handling of the product.

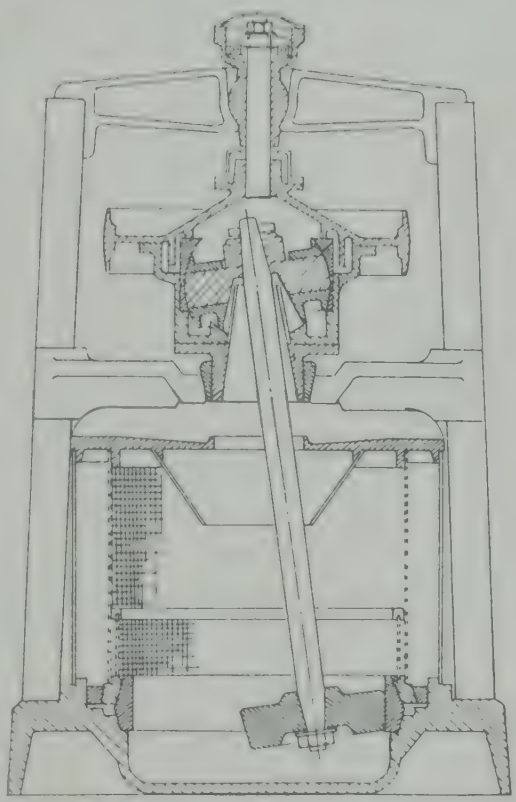


Fig. XIII-19.—Griffin Mill.

Although these medium-speed units are widely used, not nearly as much published information is available on their performance as in the case of ball mills. This is probably because the number of possible operating variables is smaller, and the mills are built to function in one way, at a fixed speed. There is nothing to prevent one of the many forms that have been devised from working on a wet-crushing cycle, but as far as is known dry grinding is always practised. Once again it is better to reduce the water content of the solid below 2%, but this is not so essential as with dry ball-milling, and in fact it has been proposed to combine the operation of an air-swept mill with a drying process by heating the air entering to any desirable figure.

* Farrant, *loc. cit.*

High-speed mills, revolving at speeds up to 5000 r.p.m., are the simplest of all grinding units. These mills rely on impact, as will be seen from the diagrams of swing-hammer mills set out in Fig. XIII–20. The central axis carries four or more loosely pivoted hammers, which dash the feed against the rigid bars of the breaking cage. A number of patents have covered special forms of both hammer and cage bars, as the wear is very rapid with a badly set machine. The bars can be reinforced by a screen to take off the fines, but are frequently used alone. The material passing the bars is a good deal finer than the setting of the bars would suggest, and a

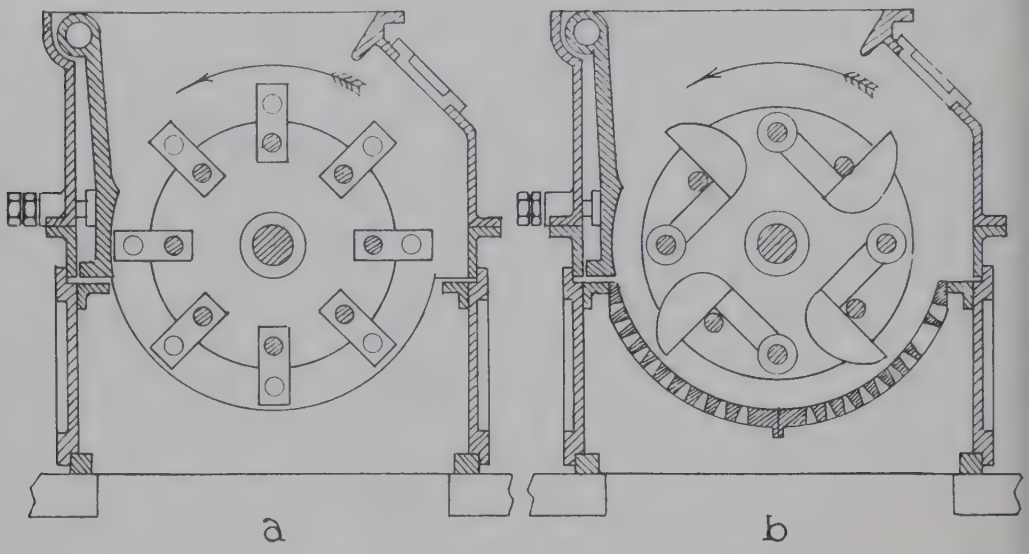


Fig. XIII–20.—Swing-Hammer Mills.

0·1-in. slit will give a product which is almost all well under 20-mesh. This is a general finding with all grinding units in which a screen is incorporated. A simple hammer mill is extremely versatile in size requirements, and can be used for breaking large rock to 1-in. pieces, reducing these to 10-mesh and finally grinding the material to a fine powder. In the case of a natural limestone a large mill breaking 8-in. lumps to 1·5 in. took 60 h.p. for an output of 30 tons/hr. This is 2·7 reduction tons per h.p. hr., quite a reasonable figure. A smaller mill, taking 15 h.p. crushed this 1·5-in. feed to a 10-mesh product at a rate of 2 tons/hr. A screen analysis of this showed that 80% passed a 20-mesh and 46% a 65-mesh screen, so that a classifying unit would have isolated up to 1 ton/hr. of fines below 60-mesh.

When very brittle solids such as dried filter-cake or crystals have to be broken up, the swing hammers may be replaced by rows of fixed bars, rotating in co-centric sets of fixed members. The feed is introduced at the centre and is flung outwards by centrifugal force through the breaking zones. All types of brittle solids, and particularly those which soften on heating, can be broken down in this way. The mill acts as a centrifugal fan, and draws in a supply of air sufficient to work a cyclone dust collector and a bag filtering unit without subsidiary air fans. These mills seem to be more costly in power than the simpler swing-hammer type. A unit grinding glue flakes from 1-in. size down to 30-mesh took 20 h.p. for an output of less than 1 ton/hr., while another larger mill required 50 h.p. to grind 3·5 tons/hr. of starch down to a 12-mesh product.

It has been suggested that the high-speed mill, in one form or another, can give a finer product than either the ball mill or the medium-speed roller mill.* With these two latter it is difficult to go below a product of screen size 200-mesh, corresponding to 0·076-mm. particle size. With a hammer mill, air-swept to provide internal classification, it is possible to produce material of 0·005 to 0·020 mm., though the power consumption may be as much as 50 h.p. hr./ton. When fillers for rubber or paint are being made, this power consumption may not be important.

While the plant described above is responsible for practically all the size reduction of importance in chemical works, one or two special methods must be briefly mentioned. Anyone who has witnessed the disintegration of a solid rock face into fragments of a few inches across by means of properly placed explosive charges will have speculated whether such a process might not be used for general size reduction.

Although, for a variety of reasons, this has not been found practicable, a logical development has been found in explosive disintegration. The material to be disintegrated is filled into large pressure vessels provided with a quick-opening valve. The vessel is put under pressure, conveniently by steam, and then this pressure is blown off as rapidly as possible, preferably by ejecting the bulk of the contents against a baffle plate. The process has been used on porous fibrous materials such as wood chips, and has had some success with brown coal.† For this latter it is at present held to be

* Berry, *Ind. Eng. Chem.*, 1946, 38, 672.
† Meigs, *Chem. Met. Eng.*, 1941, 48 (2), 122.

more costly than ball-milling. A simple type of mill which has appeared in the last few years is in essence a ball mill without balls or grinding surfaces. It consists of a wheel of 30 ft. diameter, carrying lifting bars which raise the feed to the top of the wheel periphery, whence it is released to fall on to a breaking grid and a sieve to remove the fines. Very few data are available, but it is claimed* that 10-inch lumps may be broken to 200-mesh at one operation, though it is probable that only certain brittle solids can be disintegrated in this way.†

The very fine particles produced by hammer mills are also made by the so-called "jet grinding". This process makes use of the

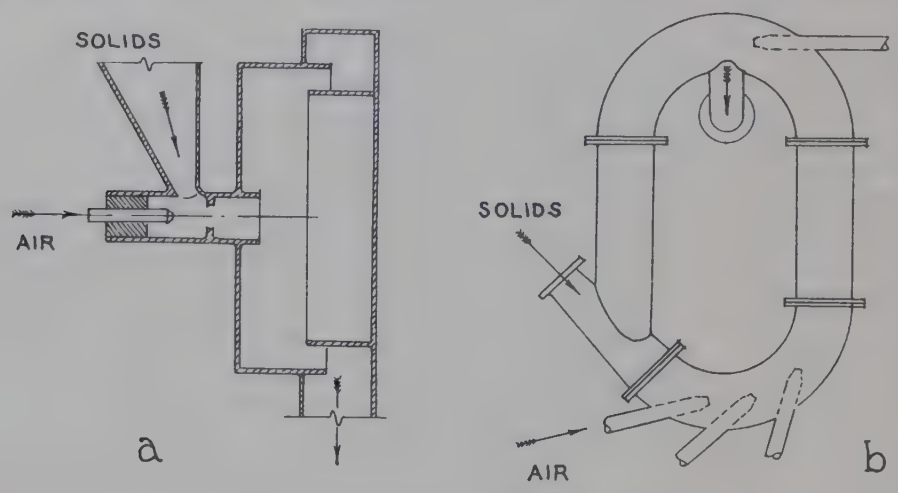


Fig. XIII–21.—Jet Grinding Plant.

kinetic energy of a stream of fluid to disintegrate particles of solid in suspension. This is achieved by flinging them against a breaker plate, as in Fig. XIII–21a. The suspending fluid is either air or steam, and pressures at the discharge nozzle of the order of 100 lb. are used. Alternatively, the suspension may be circulated for some time in a circular ring tube, as in Fig. XIII–21b, and abrasion or particle impact gives the size reduction required. The power requirements may be fully as great as those used in the hammer mill for the same type of work, but particles of less than 0·005 mm. in size may be obtained.‡

* Farrant, *Trans. Inst. Chem. Eng.*, 1940, 18, 56.
† Banks, *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1890, 1945.
‡ Berry, *Ind. Eng. Chem.*, 1946, 38, 672.

CHAPTER XIV

SCREENING, CLASSIFICATION, AND SEDIMENTATION

Screening. The grading of solid materials by size ranges is essential to many industries, and probably developed first in connection with building and road-making. As is always the case where a long tradition exists, the grading is handicapped by methods developed in connection with other purposes or in order to fit plant already in being. First and foremost among these anomalies is the position of screening. All screening or sieving figures are, or should be, expressed in terms of a standard type of sieve. Unfortunately there are at least three standard systems in use today in the English-speaking world and several more in other sections of the community. The three standard systems based on feet and inches have slight but significant differences in mesh sizes, as will be seen from the following table.

Mesh Number	Aperture Side, in Inches		
	B.S.I.	I.M.M.	Tyler
10	0·066	0·05	0·065
30	0·0197	0·0167	0·0164 (35-mesh)
60	0·0099	0·0083	0·0082 (65-mesh)
100	0·0060	0·005	0·0058
200	0·0030	0·0025	0·0029

All these screens have square apertures, and it is a pity that the actual aperture size was not taken as the basis for calibration. Then instead of a 200-mesh B.S.I. sieve, a sieve of 0·003-inch aperture would be specified, without any need for reference to a long table to see what size of particle was to pass the sieve. It is not easy, however, for the wire-weaving firms to work to a certain size of aperture rather than a certain number of threads to the inch, as in ordinary textile practice, and it is probable that screens will continue to be graded for all time on the basis of meshes per inch. It may be mentioned briefly here that a screen analysis of a granular solid will

give very inconsistent results unless the method of sifting is very narrowly specified, and it is probably better to rely on some mechanical shaking device rather than on hand agitation.

The simplest form of screen or sieve, designed to separate lumps of 1-in. size or over, is a fixed bar system (" Grizzly ") set at such an angle that the material, discharged from a rail-car or hopper, rolls down from top to bottom, with the fines dropping through the slots between the bars. The angle of slope chosen is about 30° to the horizontal, and an output of 1 ton of material/hr./sq. ft. of screen area can be obtained for each 1 in. of screen opening. This is a very approximate figure, as the output will vary widely with amount of oversize, angle of slope, and nature of the material being screened. It is true with all types of sieve that the output will be very markedly affected by any attempt to screen out all the undersize that should ideally pass through the sieve. If the efficiency of a screening operation is defined as the ratio " fines separated/fines in feed ", then normal working will not give more than 85% efficiency. Still lower figures are frequently obtained when the feed contains a large proportion of grain sizes just above and just below the actual aperture size. Any attempt to work to an efficiency of above 95% may drop the output of the screen to less than a third of the normal figure.*

The output of these " Grizzly " screens may be increased by moving some or all of the bars by an eccentric wheel. In a screen of this form, the bars are mounted on an eccentric shaft with adjacent members 180° apart, and driven so that the " high " bars are moving forward. This conveys the material gently along the screen, which can now be mounted horizontally. A screen 12 ft. long and 4 ft. 6 in. wide requires 2 h.p. for driving and will pass about 2 tons/hr./sq. ft. for each 1 in. of opening. In another form the bars are replaced by a series of interlocking discs, rotating to carry the feed forward. The power requirements are rather high—5 h.p. for a screen 6 ft. long and 2 ft. wide, but an output of 3 tons./hr./sq. ft. per in. of opening can be obtained.

These heavy screens are only intended for the separation of the largest sizes, from 12 in. down to 1 in. For a somewhat smaller grain, and particularly for the sizes from 2 in. down to $\frac{1}{4}$ in., a circular rotating screen is used. These screens, known as " trommels ", are cylinders of heavy wire cloth or perforated metal, set at a slope sufficient for the feed, entering at one end, to pass slowly down

* See, e.g., Farrant, *Trans. Inst. Chem. Eng.*, 1940, 18, 56.

the cylinder as it rotates. As will be seen from Fig. XIV–1, the trommel can be of considerable length, and several screen sizes are usually separated in the one plant. The cylinders have been made in sizes up to 30 ft. long and 7 ft. diameter, and the power requirements are relatively low. For a screen of length L ft. and diameter D ft. the approximate h.p. required is $(L \times D)/8$.

The trommel is subject to the same speed restrictions as the ball mill, with a limiting speed of $\sqrt{(1{\cdot}62/D)}$ r.p.s. This for a screen of 3 ft. diameter is equivalent to 44 r.p.m., but measurements on screening efficiency suggest that the best speed is much less than this, at 15–17 r.p.m. The issue is further complicated by solid breakage, dust nuisance (in dry screening), and wear and tear on the screens. The output of the trommel for these normal conditions is usually quoted as 50 lb./hr./sq. ft. per mm. of aperature, or 0·58

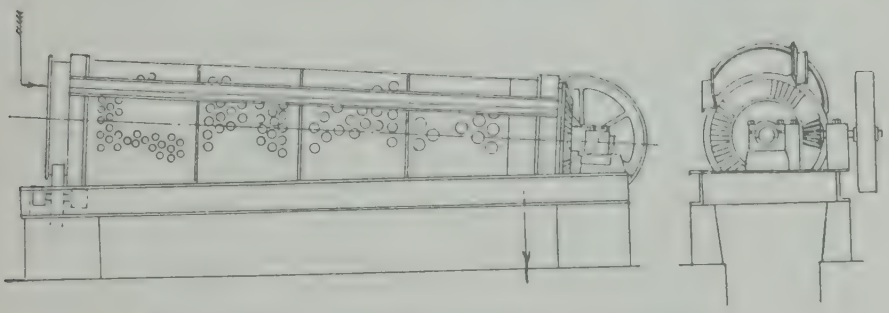


Fig. XIV–1.—Rotating Screen or Trommel.

tons/hr./sq. ft. per in. These figures can often be exceeded with free-screening solids without too much oversize (less than 50% in the original feed). Again there will be variations from one material to another and with alterations in the screening efficiency. If the trommel is used for wet screening, or with internal water-feed boxes to wash the upcoming side of the screen, the output is almost doubled compared with dry working.

In the trommel shown in Fig. XIV–1, all the feed passes over the finest and most delicate screening fabric, causing a large amount of wear. To overcome this, especially with smaller screen sizes, the trommels can be mounted as separate units, as shown in Fig. XIV–2. Conical screens are commonly used to avoid a series of tilted axes, and mounted one above the other, with the feed entering the uppermost screen. The coarse material is removed before the outer and finer screen is reached. A round section is not easily made with these cone forms, and in consequence they are often built up in

hexagonal section, with some slight increase in output. Logically, the screens, being of different diameter, should be driven at different speeds, and this is the reason why each screen is mounted quite separately, with fines from one feeding into the end of the next.

Trommels become increasingly inefficient compared to other forms of screen as the screen aperture drops below $\frac{1}{4}$ in., and the newer mechanically vibrated screens are sometimes claimed to be replacing trommels for all size ranges. This may be due in some cases to the

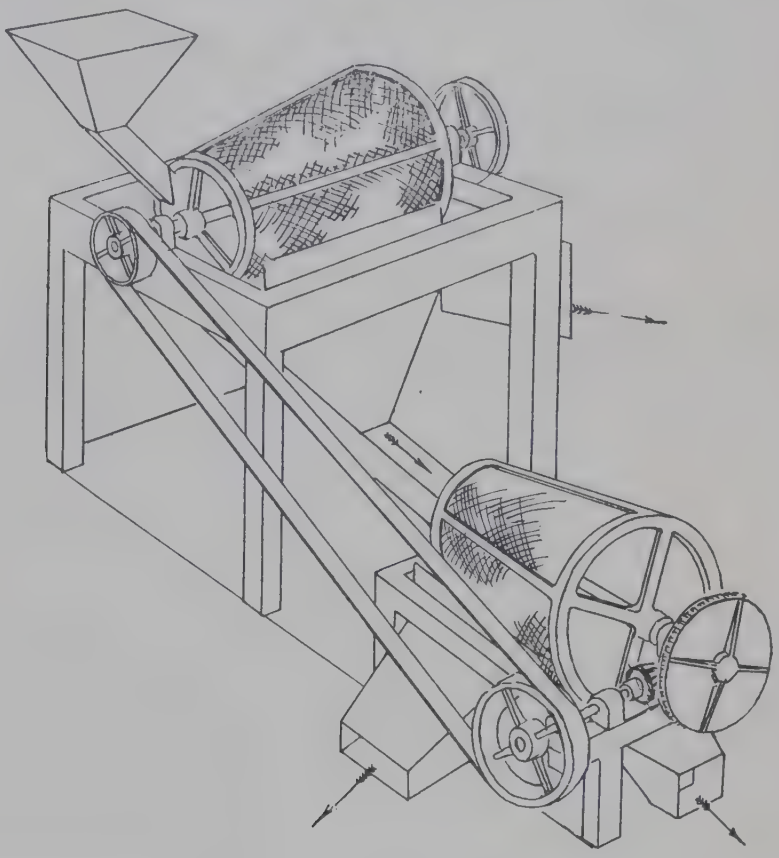


FIG. XIV–2.—CONED TROMMELS.

dust and noise which are usually associated with trommel-working. The rotating screen is easy to construct and to operate, and would appear to hold a secure place in movable grinding plant as used in road work, but it may be outmoded in chemical plants.

Shaking screens are rigid frames carrying a screening surface which can be vibrated either with the frame or separately. The screening surface is usually a square-aperture wire cloth with variations in the thickness of the wire used, and hence of the screening area as a percentage of the total surface. The stronger the wire for a

given number of meshes to the inch, the lower the true screening area. A few punched-plate screens with circular holes are made, and for very steeply sloped screens an elongated rectangular aperture is sometimes used. A screen may be shaken mechanically, usually by an unbalanced pulley, or vibrated electrically by attachment to the core of an electromagnet. In the first case the motion of each section of the screen is through some approximately circular orbit in a vertical plane, while in the second the motion is in a short straight line perpendicular to the screen surface.

In either case a free solid particle on the screen surface will move relatively to that surface at each cycle, usually by jerking into the air. In an ideal case the particle should strike the screen once in each cycle, so that a relation should exist between the velocity with which it is projected into the air and the total cycle time. Working on this assumption, R. F. Davis* suggests that the minimum speed for a screen in which the movement is a circle of radius R feet is given by $S = 1/2\pi \sqrt{g/R}$ r.p.s. The best operating speed would appear to be about twice this minimum rate. For a screen with a "throw" of $\frac{3}{8}$ in. ($R = 0{\cdot}1875$ in.) the minimum speed and the best operating speed were 435 and 800 r.p.m. respectively. In the case of an electrically vibrated screen, the motion is more complicated, as the maximum speed under the influence of the electrical field is not known. If the screen travels a distance D between the two extreme positions, and has a speed of N complete oscillations per second, the mean velocity must be $2DN$ ft./sec. Assuming this terminal velocity for the solid particle, it will be projected upwards perpendicularly to the screen surface, at an angle α to the vertical. The "time of flight" will be given by the formula $2DNT = \frac{1}{2}gT^2 \cos\alpha$, and the displacement on the screen caused by this will be $\frac{1}{2}gT^2 \sin\alpha$. For a screen movement of 0·125 in. and a rate of 50 cycles/sec., where the screen is sloped at 10° to the horizontal,

$$T = \frac{0{\cdot}25 \times 50}{12 \times 16{\cdot}1 \times 0{\cdot}9848} = 0{\cdot}066 \text{ sec.}$$

also $\quad \frac{1}{2}gT^2 \sin\alpha = 16{\cdot}1 \times 0{\cdot}00435 \times 0{\cdot}1736 = 0{\cdot}0121$ ft.

This is equivalent to movement across the screen at a rate of 2·2 in./sec., with a possible 15 impacts on the screen at the same time. This analysis takes the simplest possible view of the case, as

* *Trans. Inst. Chem. Eng.*, 1940, 18, 76.

the screen and load may vibrate in a number of ways, certain of which may actually check the rate of screening.*

In the case of mechanical vibration it is not necessary to fix the screen in an inclined position, though this is generally done, but with electrical vibration some screen slope is necessary. Ideally, a fragment could fall through each aperture at every stroke of the screen, but this is a practical impossibility. A large proportion of the solid strikes the partition wires between the holes, and the proportion of the total screen area covered by these partitions must increase as the strength requirements grow more onerous. Again, as the percentage of undersize in the mixture on the screen diminishes, the number of apertures blocked by oversize at each stroke increases, and ultimately the rate of passage of the undersize becomes very small. Normally, when a mixture of small and fine grains is shaken, the fines flow to the bottom of the mixture, and the coarse fragments form a layer on the surface, thus increasing the rate of sieving. If the agitation is too violent, or the solid forms a very deep bed on the screen, this stratifying may be interrupted. Again, the solid on the screen should be dry and free-flowing, or wet enough to flow as a thin slurry. Some types of material, such as magnetic iron ores, tend to stick together on the screen, while other powders flow freely even when a small proportion of water is present.

It used to be general practice to use relatively slow mechanically operated screens for the separation of particles down to 50-mesh B.S.I. (0·012 in.) and to turn to electrically vibrated sieves for the finer sizes. These slow (2–400 r.p.m.) screens are now being replaced by mechanically vibrated screens with a frequency of 1500 cycles/min., and the latter are competing against electrically vibrated plant for all screen sizes.

A mechanically operated screen of the form in which the " cloth " only is vibrated is shown in Fig. XIV–3. An eight-pointed cam is driven at 200 r.p.m., thus giving 1600 strokes/min. to a plunger, with an amplitude of $\frac{1}{8}$ in. It would appear that this type of vibration approximates closely to the electrical movement discussed above. The screen cloth recommended appears to be above 50-mesh in size, so that the screen may not be very effective for smaller sizes. An alternative design, in which the vibration given by an unbalanced flywheel is transmitted to the screen frame, is shown in

* Göbel, *Braunkohle*, 1941, 40, 581.

Fig. XIV–4. It will be seen that the whole screen hangs freely by a rope suspension from the four corners, so that vibration is confined

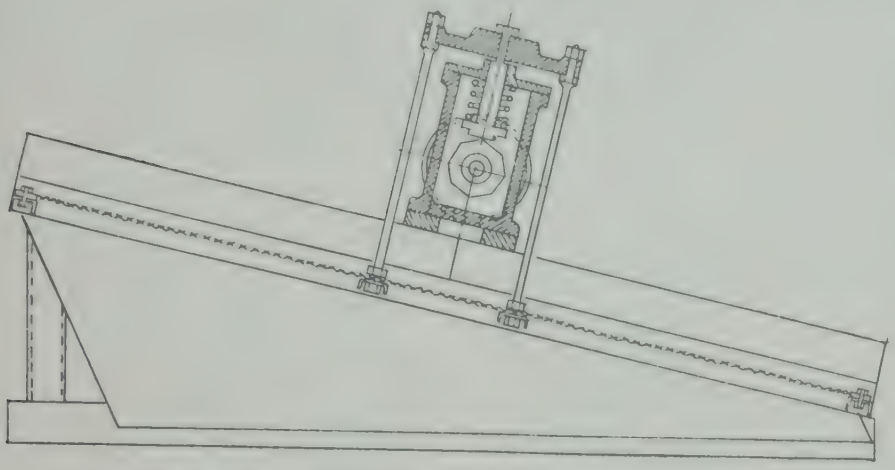


Fig. XIV–3.—Vibrating Screen—Mechanical Action.

to the screen and does not extend to the surroundings. The screen is sloped, but the movement of the material can be adjusted by altering the slope, or even by driving against the slope. The speed

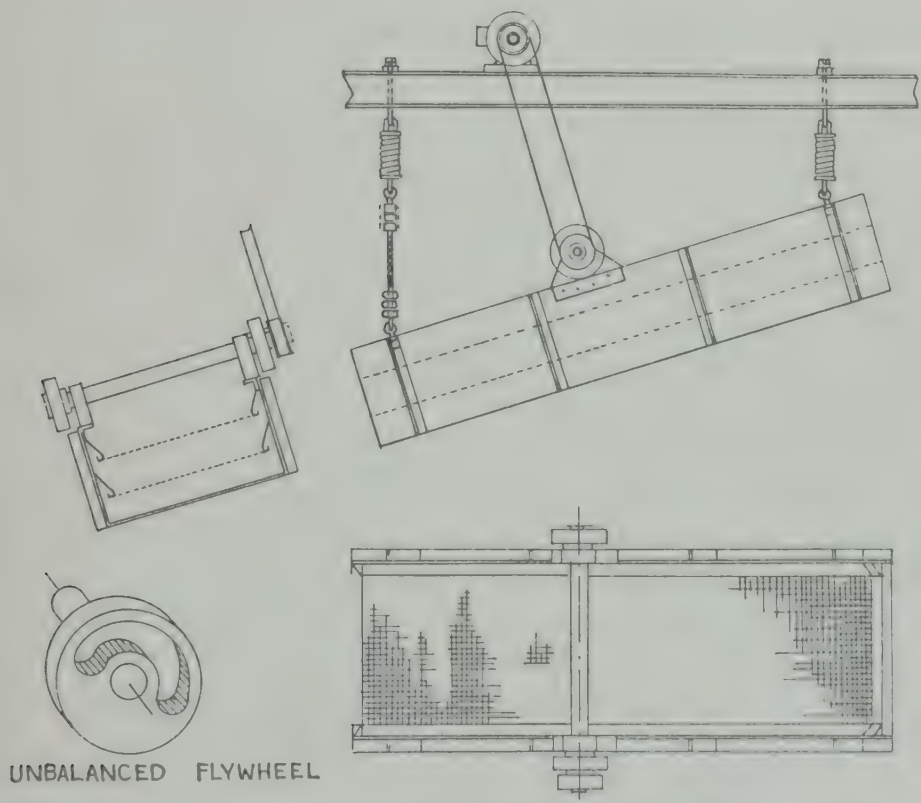


Fig. XIV–4.—Vibrating Screen—Mechanical Vibration.

of drive of the flywheel is from 1000 to 1500 r.p.m., and the frame carries two sieves or " decks ", thus splitting the feed into three fractions. A frame 4 ft. wide by 8 ft. long requires 2 h.p. for driving at 1200 r.p.m., and the eccentricity of the motion can be varied by adjusting the unbalance of the pulley-flywheel. This power figure

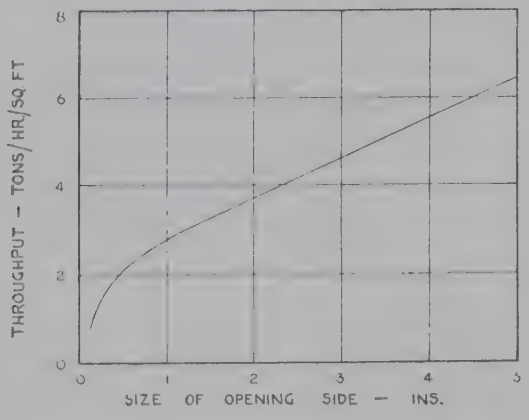


FIG. XIV–5a.—SCREEN OUTPUT CURVES.

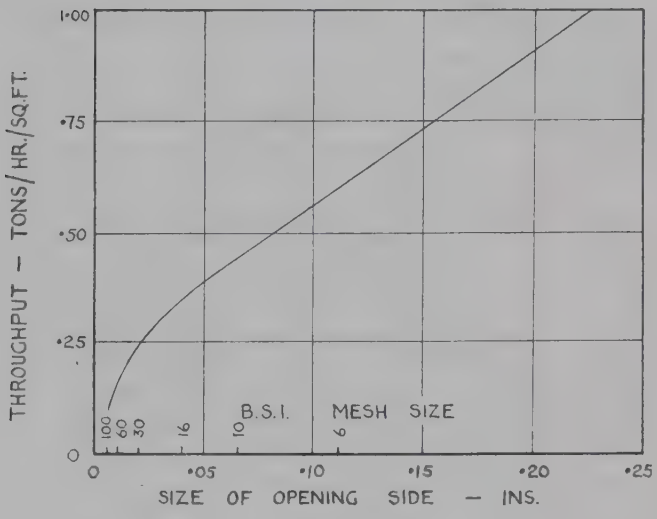


FIG. XIV–5b.—SCREEN OUTPUT CURVES.

seems to be on the low side, as similar makes with only slight variations are said to use up to 5 h.p. for the same screen area.

The output of these screens depends in the first place upon the screen aperture. The curves of Fig. XIV–5a and 5b show the throughput of fines from a free-screening feed containing about 40% of oversize and a normal spread of sizes in both residue and product. These can be widely modified by the solid being screened; thus coal gives only 70% of the throughput obtained with gravel. If a large

proportion of the particles are very close to the aperture size (from 0·75 to 1·5 L, where L is the aperture), then the output is reduced, though the figures given in the curves are reasonably correct for some 30–40% in this region. The screening efficiency for the data of Fig. XIV–5a and 5b is about 90%, and output will vary with this efficiency as already indicated. A number of formulae have been proposed to cover some or all of these factors, but no generally accepted equation has yet been established. The most important common factor in all published figures is the way in which screen capacity drops as the aperture decreases below about 100-mesh size (0·006 in.).

The choice between a vibration transmitted to the whole frame and one in which the cloth only is shaken is debatable. If the cloth is shaking relatively to the frame, it is not easy to avoid fatigue at the points of attachment. The cloth must be made from special alloys designed to resist these stresses, and special methods of fastening used to minimise the local stress. Even with these precautions wear and tear are considerable. In one report a stainless-steel cloth in a typical case had a life of only 33 days, while an ordinary steel cloth failed in 40 hours.* On the other hand, the screen-shaking mechanism is more powerful and tends to transmit vibration to a greater extent. It is possible that certain materials are particularly suited to cloth vibration and others to frame action, but the tendency appears to be towards units in which the whole screen and frame are vibrated.

As an alternative to the single fixed vibrating mechanism, a number of tapping devices can be used. Thus a series of hammers mounted on the cover of a screen can deliver sharp taps on pins connected to the screen cloth below. The rotation of the activating cams is so timed that the cloth is never set in vibration as a whole but kept in a number of rapid local oscillations. Power requirements for these screens are a little lower than for the vibrated types, and the throughput appears to be very much the same. These local vibrations are said to be particularly desirable when the material being sieved tends to bind together into a self-supporting mass.

Electrically vibrated screens are said to be more economical in power than those operated mechanically. Farrant (*loc. cit.*) claims that up to four times as much power is used in mechanical vibration for screens of equal area, but manufacturers' claims do

* Farrant, *Trans. Inst. Chem. Eng.*, 1940, 18, 56.

not show so wide a discrepancy. As against this economy in power, electrical units are apt to clog (" blind ") when used with material of near aperture size, especially with solid particles that do not screen easily. The output of free-screening material is said to be higher with electrical vibration, but this is a debatable advantage in view of " blinding " possibilities. The mechanism may be totally enclosed, a great advantage with a dust-laden atmosphere, and with few moving parts needs much less attention than mechanically vibrated screens.

Vibration can be transmitted to the whole screen or only to the cloth, as in the mechanical units. In Fig. XIV–6 a screen is shown

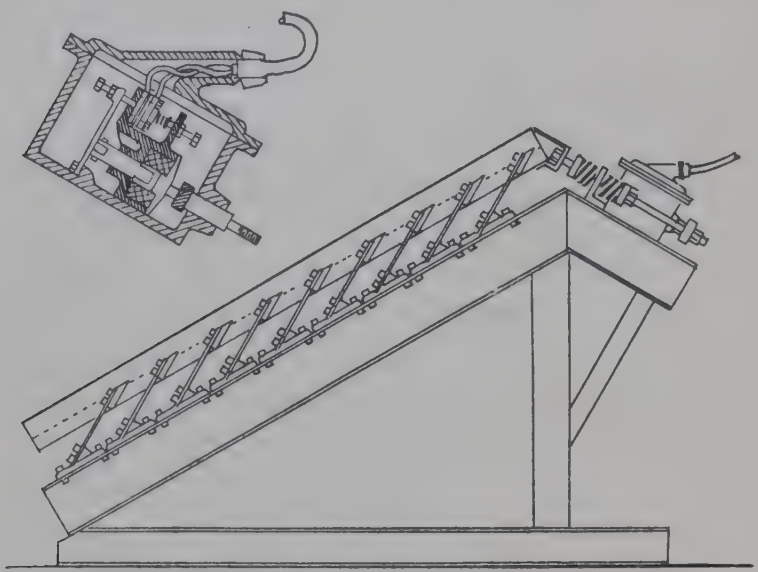


Fig. XIV–6.—Vibrating Screen—Electrical Vibration.

with a series of wooden (hickory) springs connecting it to a fixed base, with an electromagnet device moving the screen at rates up to 3000 cycles/min. The slope of the screen is about 35° to the horizontal, as against 20° for mechanical units, and the vibration has the largest amplitude at the upper feed end. This is the portion of the screen where the greatest rate of screening can be obtained, and it is claimed that this variation in amplitude decreases the tendency of certain materials to form a mat. As an example of the vibrator directly attached to the cloth, the very well known " Hummer " screen may be instanced. This, as shown in Fig. XIV–7, has the armature connected to the centre of a cloth which is usually 3 ft. wide and up to 5 ft. long. The speed is normally about 1800

cycles/min. The vibrating armature takes from 0·75 to 1·5 h.p. to drive, according to the duty required from the screen. It is common practice to use cloths with rectangular slots in these screens, as they are claimed to be less liable to blockage. A stainless-steel screen, with 10-mesh by 41-mesh apertures, gave steady throughputs of 0·5 tons/sq. ft./hr., with no " blinding " and no appreciable wear over a 15-day run. This was wet screening, with spraying, and an ordinary steel cloth choked with rust in a few hours, while a square-mesh cloth clogged very quickly.

Even though it has been claimed that electrically vibrated screens are better than other types for the separation of fine particles, there

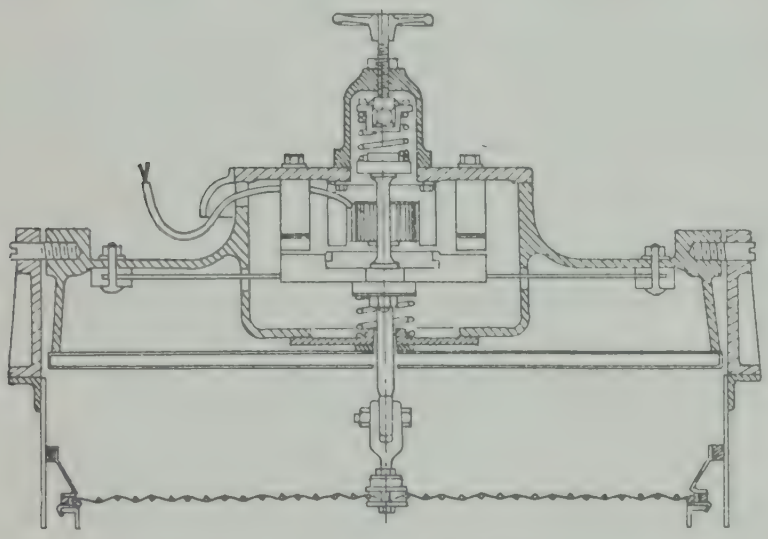


FIG. XIV–7.—" HUMMER " SCREEN—ELECTRICALLY VIBRATED.
(Courtesy of International Combustion, Ltd.)

appears in practice to be little difference in the low output when dealing with particles of below 100-mesh. The use of water and of " wet " screening does not make any improvement. In fact, a fall in output may be caused through surface-tension effects unless the screen is backed with some diaphragm to break up the water film on the outlet side. Some criticism has lately been made with regard to these conclusions, and a comparison of three grinding plants, making 100–200 mesh fines, when using (*a*) fluid classifiers, (*b*) vibrating screens, indicated an improvement in the latter case.* It is possible to increase the output of fine screens by introducing

* Reed, *Amer. Inst. Min. Met. Eng., Tech. Publ.* 1820, 1945.

two different types of vibration. In one case a wooden comb drawn over the screen at 10-minute intervals increased the output by 50%, while the author has seen sugar-sifting machines in which small rubber balls were bounced over the screens at intervals. A special form of screen has been devised for materials such as flour which have to be brushed through the apertures, but these plants are unusual in the chemical industry.

It is possible to combine the action of a screen with that of a conveyor, and a number of these have been built. The Undulo screen for coke consists of a perforated rubber band, moving above a series of wooden paddles which give a wave-like motion to the band. The coke is conveyed over a series of hoppers, each receiving an appropriate size range of coke, and little power is required, with no breakage and little dust.

The foregoing paragraphs have described only a few of the large number of screening devices that have been built and operated in industry, but present some idea of the operation and output of typical plant.

Classification. The underlying principle of fluid classification is the balance between gravitational (or centrifugal) acceleration and the resistance to motion offered by the fluid to a solid particle moving in it. When a spherical particle is moving slowly in a viscous fluid, the displacement of fluid around the solid is small and layers of the medium slide over each other in viscous flow. Then motion is in accordance with Stokes' Law, and the resistance offered by a fluid of viscosity η to a sphere of diameter D moving with a velocity V will be given by

$$R = 3\pi D\eta V$$

As the velocity increases, due to gravitational acceleration, the motion tends to lose streamline character and become eddying. When fully turbulent conditions are established, the resistance becomes

$$R = kD^2\rho V^2$$

where k is some constant (approximating to $\pi/4$) and ρ is the fluid density.

A single spherical particle, in free fall in a fluid of density ρ, and having itself a density S, will have an effective weight of $\frac{1}{6}\pi D^3(S - \rho)$. This weight will act downwards with a force equal to $\frac{1}{6}g\pi D^3(S - \rho)$ and cause an increase of velocity until the accumulated resistance

becomes equal to the gravitational force. This may be either under the conditions of viscous or turbulent flow. In the one case

$$\tfrac{1}{6}g\pi D^3(S - \rho) = 3\pi D\eta V$$

or

$$V = \tfrac{1}{18}\, gD^2 \frac{S - \rho}{\eta}$$

In the other

$$\tfrac{1}{6}g\pi D^3(S - \rho) = kD^2\rho V^2$$

or

$$V = \sqrt{\left[\tfrac{1}{6}\frac{g\pi}{k}D\left(\frac{S - \rho}{\rho}\right)\right]}$$

These two expressions may be simplified to the general forms

$$V = K_1 D^2\left(\frac{S - \rho}{\eta}\right) \text{ and } V = K_2\sqrt{\left(D\frac{S - \rho}{\rho}\right)}$$

where K_1 and K_2 stand for numerical constants. A similar relation must hold where the fluid is moving and the particle is stationary,

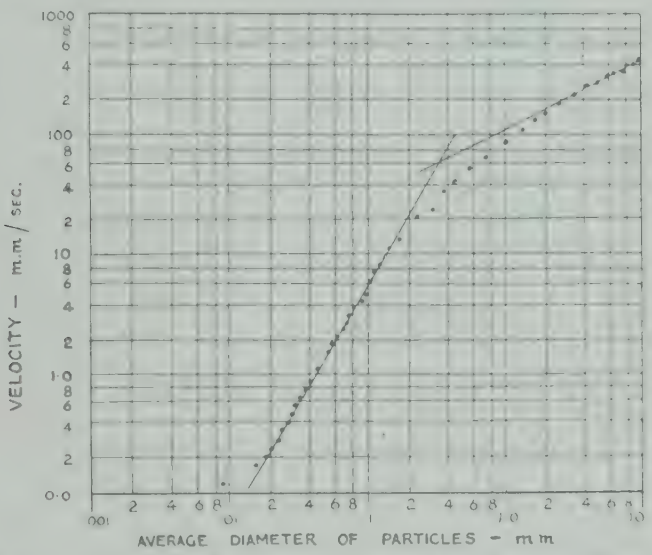


Fig. XIV–8a.—Falling Velocities: Quartz Particles in Water.
(*After Stewart, "Trans. Inst. Chem. Engr."* 1933, 11, 124)

and there is, therefore, for any fluid-solid system a limiting relative velocity to which the particle may attain in free fall, or which the fluid may reach before the solid is lifted. The only difference between spherical or irregularly shaped fragments is that the constants in the equations will be altered. The "ultimate falling velocity" for solid particles of the same material and form but of different sizes will be proportional either to D^2 where D is some characteristic dimension of the particle, or to $\sqrt{D}$ where the velocity is high enough for the flow to be turbulent. In Fig. XIV 8a the

falling velocity of quartz particles ($S = 2{\cdot}65$) in water is plotted against a linear dimension. It will be seen that Stokes' Law applies for velocities below 6 mm./sec. with a particle of below 0·1 mm. (150-mesh). After an intermediate region full eddying flow conditions hold for particles of above 1 mm. (16-mesh) and velocities above 100 mm./sec. For quartz particles in air a similar plot, shown in Fig. XIV–8b, indicates viscous flow up to 200 mm./sec. and a particle size of 0·075 mm. (200-mesh). Turbulent conditions, sometimes described as governed by Oseen's Law, hold for velocities over 1000 cm./sec. and particles of more than 2 mm. in size.

The figures given above apply to single solid particles in free fall.

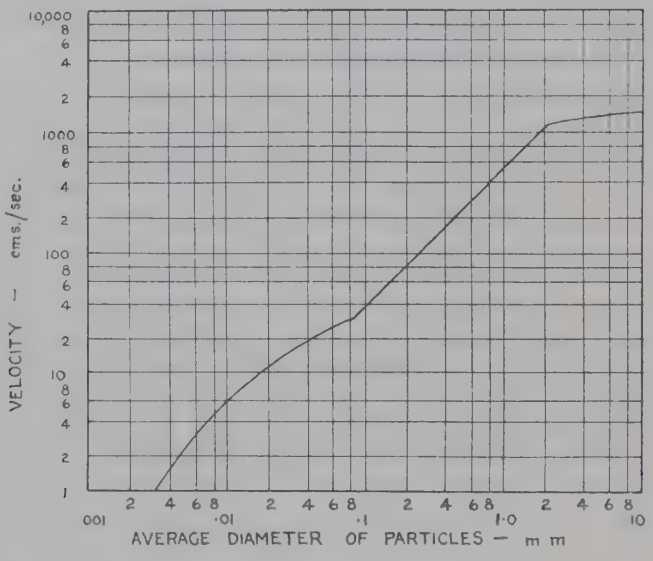


Fig. XIV–8b.—Falling Velocities: Quartz Particles in Air.
(*After Martin, "Trans. Cer. Soc.,"* 1926, 26, 21.)

In all industrial classifications very large numbers of fragments in a limited volume of fluid are being treated. These particles will interfere with each other to a degree which steadily increases as the size of the individual fragments decreases and the solid concentration increases. It is not possible, therefore, to apply the actual rates of fall determined for individual particles to the design of classifiers. Relative rates of fall of reasonably even-sized particles are affected to the same extent, however, and the ultimate falling velocity remains the ultimate basis of classifier practice. It must also be noted that small heavy particles have the same falling velocity as larger lighter fragments. This makes it impossible to carry out size separation by classification when two materials are present.

Certain minerals also tend to break into shapes which make for easy lifting by an upward-streaming fluid. Mica in quite large flakes can be elutriated from felspar fragments of smaller dimensions but rounded form, despite the fact that both materials have approximately the same solid density.

When small particles are involved, rates of fall are small, even in air, and the separation in space between two unequally sized pieces is hardly large enough at the end of any reasonable time to base a classifying process on it. Thus for a solid fragment of 300-mesh size (0·053 mm.) the free-falling velocity may be 12 cm./sec. in air, while for another fragment of 240-mesh (0·066 mm.) the corresponding figure is 20 cm./sec. The distance between the two after a free fall for 1 sec. cannot be more than 8 cm. and owing to hindered fall will be much less than this. If it were possible to increase both the ultimate falling velocities by some positive factor, the " gap " at the end of a definite period of fall would be increased. When solid particles are spinning in a circle the effective weight in an outward direction—across the fluid stream which holds them in suspension—will be their normal weight multiplied by the centrifugal effect. This for a fluid stream of velocity V moving in a circle of radius R will be V^2/Rg. Then substitution of this modified weight in the formulae given above shows that the ultimate falling velocity will be increased by the factor V^2/Rg when the movement of the solid is governed by Stokes' Law, and by the factor $\sqrt{V^2/Rg}$ when motion is turbulent. If, therefore, particles are spinning in an air stream moving in a circle of 1 ft. diam. at a velocity of 100 ft./sec., the velocities are increased by the factor $100^2/(0{\cdot}5 \times 32{\cdot}2)$, or 620 in the first of the instances above. The gap at the end of 0·03 sec. between two fragments starting from the same point, one of 240- and the other of 300-mesh, is now up to 144 cm. or nearly 5 ft. This is in the time that the air stream takes to pass once round the circular path, and it is clear that a much more effective process for separation is being used. This improvement is the reason why nearly all air classifiers use centrifugal methods, but the increasing differentiation means increased power charges. Where water is the classifying fluid this power consumption has definitely checked the application of centrifugal principles, and the low power requirements of simple settling methods, with water as the fluid, have led to a wide adoption of these methods despite the greater apparent efficiency of air classification. There is, of course, no reason why

other fluids than water or air should not be used for classification, apart from purely economic factors.

Liquid Classifiers. The simplest form that these can take is the settling tank, in which the larger fragments settle and the fines overflow. Such tanks are upward-current classifiers, as can be seen from the following argument.

Consider a tank of cross-section A, into which a slurry flows at a rate of Q cu. ft./min. It may be assumed that movement is confined to the top T feet of the tank. Then the volume of slurry in motion is AT cu. ft., and the time taken for any unit volume of slurry to pass through the tank will be AT/Q. If a solid particle is to settle out in the tank it must fall out of the active top layers in this time. That is to say, it must fall T ft. in a time AT/Q, or have a settling velocity of Q/A ft./min. Thus the settling tank will tend to remove all fragments having an ultimate falling velocity of Q/A ft./min. or more, an operation which could be paralleled by an upward current of water of similar velocity.

When this settling tank conception is developed along obvious lines, it will be realised that the bottom should be drawn out into a cone for ease of removal of the settled solids, while for ease of fabrication the top would also be circular, making the tank into a single large cone. The feed would enter at one side of a radial baffle, and the fines would overflow at the other. Such tanks were in fact the earliest form of classifier, but are now only used for a very rough separation of fines, the so-called "de-sliming" process, or for de-watering. A cone 8 ft. in diameter could remove about 90% of the water from a 15% solid clay suspension, but most of the material of finer size than 200-mesh escaped with the liquid overflow. The working rate was 1150 tons of feed slurry per day, or, at 210 gal./ton, some 27 cu. ft./min. As the maximum surface area of the cone is 50 sq. ft., the effective upward velocity is 0·54 ft./min., or 2·75 mm./sec. This is the ultimate settling velocity of a particle of 0·06 mm. size (approx. 240-mesh), so that the agreement between theoretical and practical result is reasonably satisfactory, in view of the various conflicting factors.

Separation in a simple cone of this form must be far from clean-cut, owing to the carrying down of fines from the body of the slurry by the coarse material. In newer forms of mechanical classifier this is overcome by the use of rakes to turn the settled solids over, while water jets remove the exposed fines. The trough classifier is the most

widely used and best known of this type. It consists of a long narrow trough (Fig. XIV-9), set at a slight angle to the horizontal, so that the base is covered with liquid for about two-thirds of the

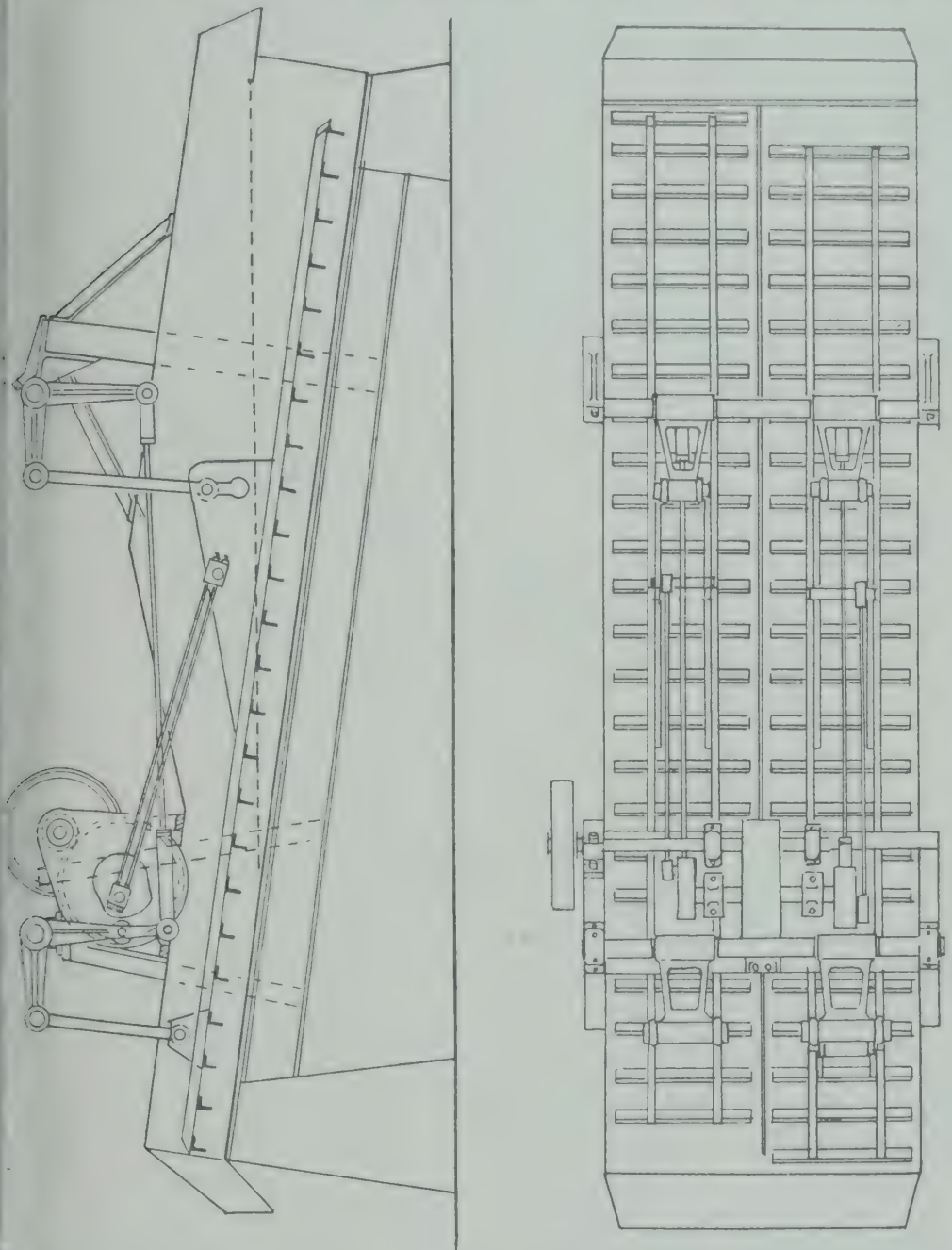


Fig. XIV-9.—Trough Classifier.

length. The feed enters at the middle of the liquid pool, and the moving rakes are set to drag the settled solids slowly up the trough, out of the liquid, through a spraying zone, and finally over the head of the classifier. Fines are carried with the liquid overflow past the

weir at the foot of the trough. Operation is not quite so simple as is suggested by this description. The main problem in classification, as in sifting, lies in the near size particle range. The action of the classifier sets up a series of zones as suggested in Fig. XIV–10. Here Zone A represents the settled solids, where movement is exclusively by the action of the rakes, and in which the finer particles are slowly disengaging themselves. Zone B is the most effective classifying section, with liquid currents and rake motion combining to establish a suspension in which the size of the solid particles increases slowly from top to bottom. Zone C is essentially a simple settling section, in which the larger fragments settle into Zone B, while the fines pass direct to the outlet, being reinforced near the weir by finer fractions from Zone B. When the classifier is over-

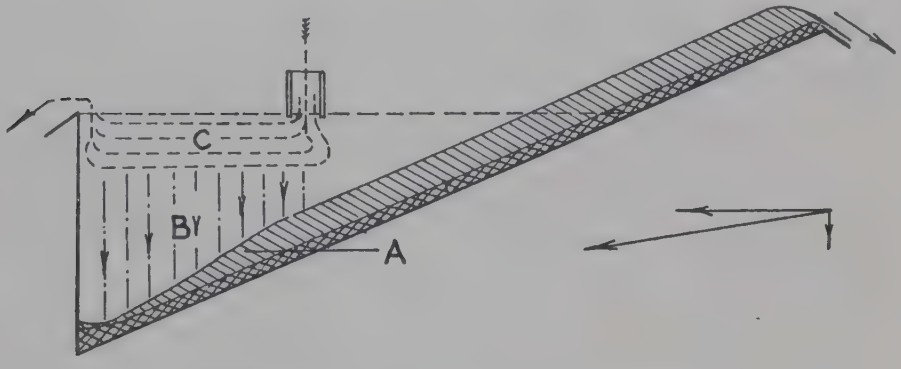


Fig. XIV–10.—Zones in Classifier.

loaded or the slurry is too dense, Zone B tends to increase in relative volume and will then discharge over the weir in a series of surges. This phenomenon can also be caused by a feed which contains an unduly large proportion—above 50%—of solid which is within 10% of the size aimed at for separation. Other makes of classifier have been devised in which the rake movement is set to check this surging tendency. The Stokes double-acting model has two sets of rakes, set like the pistons of a double-acting pump, and giving almost continuous raking action.

It is difficult to equate volumes to cross-section of the liquid pool and to obtain an indication of separation size because of the upsurge from Zone B, suggested in Fig. XIV–10. The only clear guide to performance is given by actual operating figures, such as those tabled by Taggart.* The capacity of a trough classifier of the type

* "Ore Dressing".

described above is given as 200 tons of sand (coarser than 100-mesh) and 50 tons of fines per foot of width per day. For a machine 6 ft. wide—and 18 to 20 ft. long—this would be an output of 12·5 tons of fines and a corresponding amount of sand per hour, or enough for one of the largest ball mills made, consuming perhaps 500 h.p. The power for the classifier would be less than 5 h.p., even allowing for the cost of pumping water. This low power figure is due to the slow speed of raking, which is about 12 strokes/min. for 100-mesh material. When the material is being ground wet, the water must be supplied in any event, and the slow motion allows of quite large particles being handled—up to 20-mesh—without any danger of the rakes jamming. Nevertheless these rakes form a complicated and, by

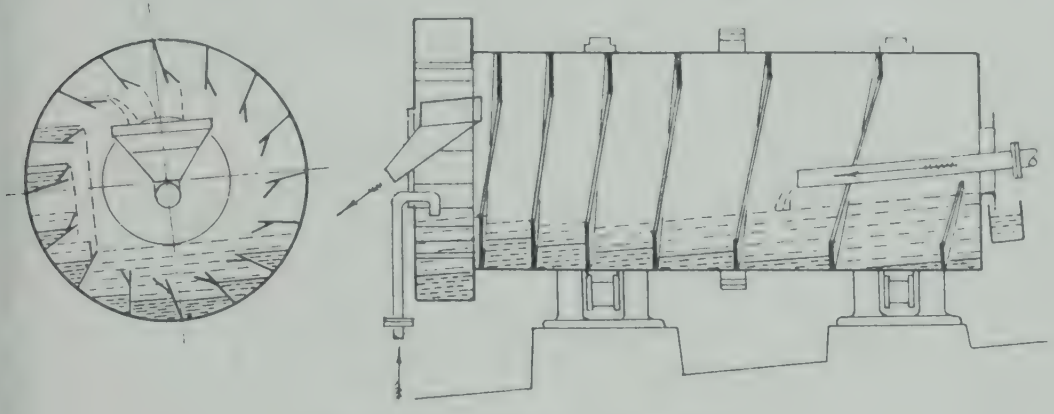


Fig. XIV–11.—Spiral Classifier.

implication, costly mechanical system, and attempts have been made to substitute a revolving spiral or Archimedean screw shell. In the classifier of Fig. XIV-11, a continuous spiral scraper conveys the settled solids towards the upper end of the cylindrical shell against the currents of wash-water and the feed entering at the middle of the unit. A classifier of this form, 6 ft. diam. by 14 ft. long, will take about 3 h.p. to drive at 1 r.p.m., while separating 10 tons of fines below 65-mesh from 85 tons of oversize every hour.

When all the solid material to be handled is around 100-mesh size or less, it is customary to use a bowl classifier. These are shallow saucer-like vessels, with a stirrer which gradually draws the settled oversize to the middle, and at the same time breaks down the pseudo-solid structure which may often be formed in mixtures of finely divided solids with a liquid. Very fine particles have certain

peculiar properties and can no longer be regarded as free settling solids. There is a tendency for the mixture to form "flocs", or agglomerated masses of particles with a rapid settling rate. This may be deliberately induced in the closely related fields of sedimentation and clarification, but may hinder accurate separation at low size ranges. In bowl classifiers liquid movements are slow, the movements of the rakes are gentle compared to trough units, and in consequence the power requirements are very small. The largest bowls of up to 28 ft. diam. take only 3 h.p. for driving. The bowl classifier can be used independently or mounted on the fines outlet of a trough unit. A combination of this type separated 17 tons/hr. of fines from a circulating load of four times this amount, making a cut at 120-mesh, and requiring 10 h.p. for the trough rakes and 2 h.p. for those of the bowl. The advantage of a combination of this kind over the simple bowl is that material of a wide size range can be handled effectively.

As mentioned above, heavy power charges have checked the introduction of the centrifugal principle into liquid classifying. One or two attempts have been made, which are referred to in the chapter on Centrifugal Practice.

Air Classifiers. It would appear that all industrial air classifiers utilise a centrifugal principle. This is due to the need for reasonably small plant and short times of operation. The simplest design is typified by the Raymond direct classifier, customarily mounted on top of a number of air-swept mills. Air sweeping through the mill entrains the solid particles of near separation size and the suspension passes up through the outer cone (Fig. XIV–12) to enter the inner cone at the ports X, X. These ports are partly covered by moving vanes, which direct the gas stream into a tangential swirl. Adjustment of the vanes gives a choice of tangential velocity, and as this increases, the centrifugal effect increases settling rates. The air stream, making one or more circuits of the central cone, leaves at the central outlet, carrying with it all the fines which have not been flung out by the centrifugal swirl. The adjustment of the vanes is so delicate a method of regulation that it has been claimed for a specific setting that all particles larger than 195-mesh are rejected, with everything finer than 205-mesh passing out with the fines. The reject oversize will undoubtedly entrain some fines and it is doubtful if the last half of the above claim could be sustained. The oversize filters down the sides of the inner cone and is returned

to the grinding unit. When working in combination with a normal circuit, the air current carrying the fines passes to a cyclone precipitator, where the solid, with the exception of very fine dust of below 0·01 mm., is thrown down. The air stream is returned through a fan to the mill, except for a portion which is drawn off to waste through bag filters. This last precaution checks the build-up of very fine dust or of moisture in the grinding classifying process.

The pressure drop across such a classifier, which determines the power requirements, is not easy to assess, as the solid must be

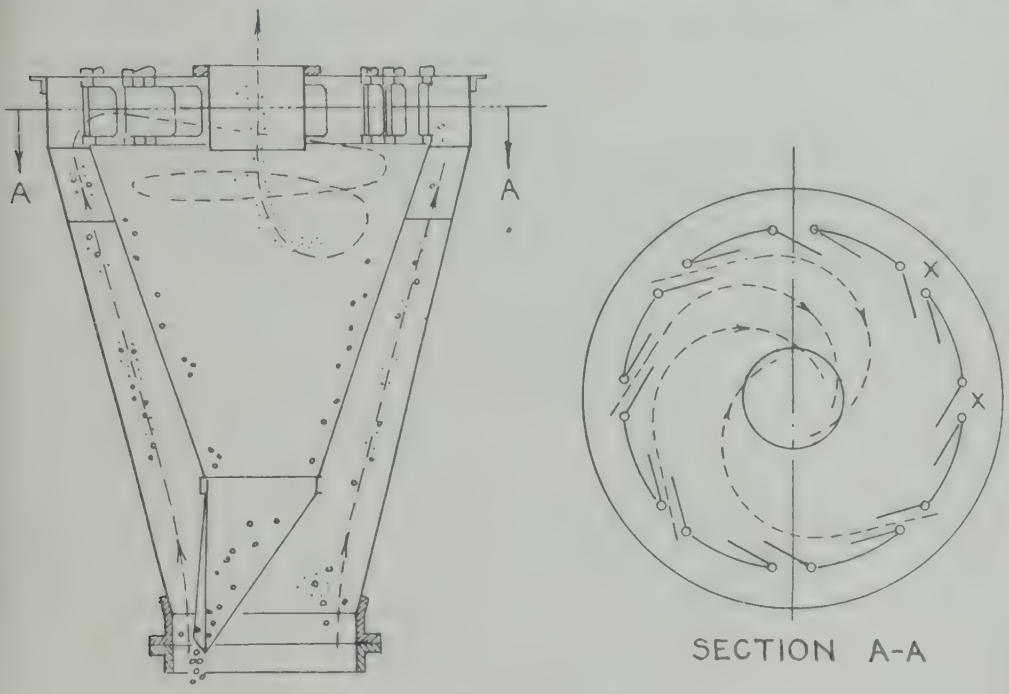


Fig. XIV–12.—Raymond Air Classifier.

(Courtesy of International Combustion, Ltd.)

removed from the mill in any case. A somewhat tentative figure for a 3-ft. diam. plant would be 5 h.p., but the throughput would depend upon the nature and fineness of the material being handled. Figures in catalogues of 3 cwt. hr. of ebonite (passing 120-mesh) and 30 cwt. hr. of lime (passing 100-mesh) illustrate this point. The amount of oversize, where this falls directly back into the mill, would clearly be almost impossible to determine.

A general review of published figures suggests that the direct classifier in its several forms is more costly in power than a mechanical unit, in which the solid is given initial velocity by some mechanical means, such as a spinning disc. This spreads the

solid feed in a fan across a rising air stream which lifts the fines, leaving the larger fragments to fall to a collector. In the air separator of Fig. XIV–13, the feed distributor A throws the solid down into a whirling air stream set up by the baffles B and the suction fan C. The oversize is re-scoured by the air entering the inner cone at D, and is finally discharged at the side spout shown.

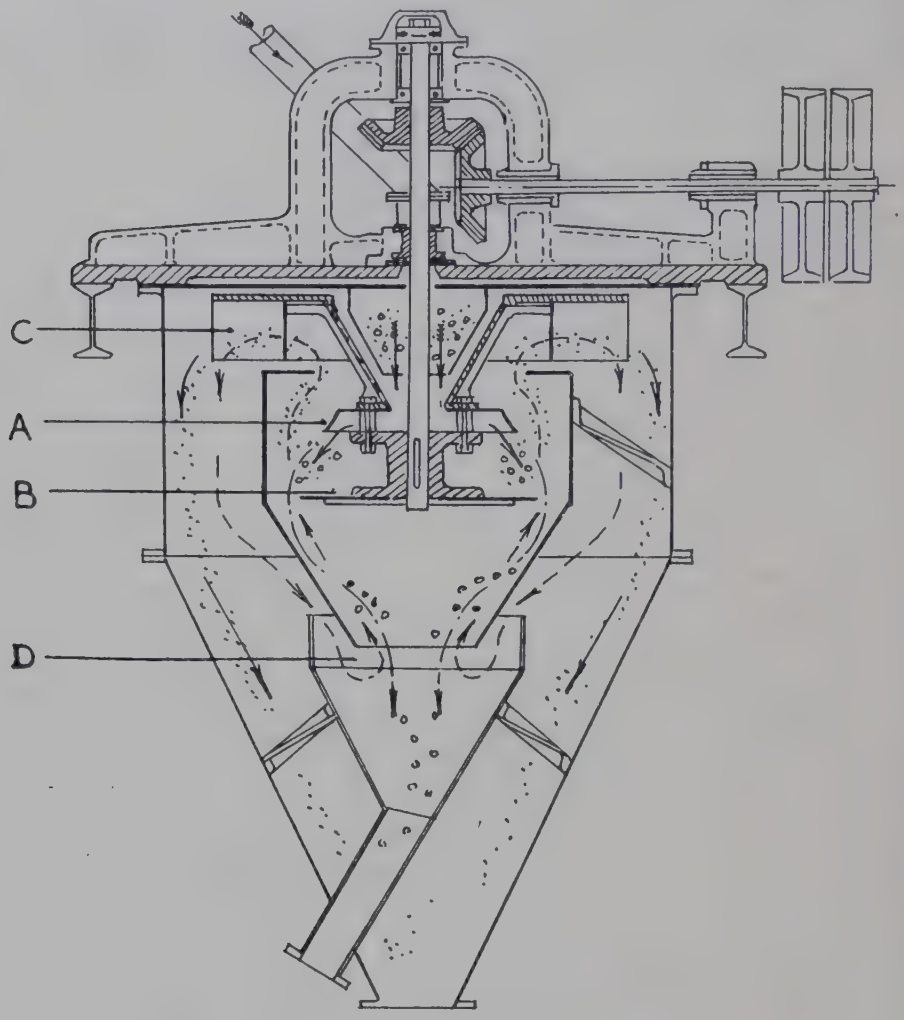


Fig. XIV–13.—Whizzer Air Classifier.

The fines carried out through the fan are deposited by centrifugal eddies at the sides of the outer cone and fall to the bottom central outlet. There appears to be little trouble due to build-up of " near " sizes, but where the feed is wet it may be necessary to bleed-off a portion of the air at intervals.

There are a number of variations on the design set out in Fig. XIV–13, incorporating different methods of air entry or of solid

distribution, but they do not appear to have any particular advantages, and the power consumption and throughput of all models is very much the same. These classifiers can be built in very large sizes—up to 18 ft. diam.—with correspondingly high throughputs. A 14-ft. diam. separator will give 12 tons/hr. of limestone passing 100-mesh, and 5 tons/hr. of 200-mesh material. It should be noted that the capacity falls with graphite to 1·5 tons/hr. of 200-mesh fines. The power requirements at these throughputs are given rather vaguely as 25–40 h.p., but other figures (makers' catalogues) are somewhat lower than this. It has been suggested that a 14-ft. diam. unit requires 15 h.p., with an output of 10 tons/hr. of 200-mesh material. Although these figures show some improvement on the performance of the direct classifier, they are still well above those quoted for a wet classifier. It may also be noted that the output is still proportional to particle size, and under comparable conditions an increase in fineness from 140- to 300-mesh will cut the output of a 12-ft. machine from 2 tons/hr. down to 0·75 tons/hr., while the power required remains constant at 20 h.p.

Sedimentation. The plant designed to produce a clear or nearly clear overflow from a liquid–solid slurry could consistently be dealt with under Filtration, but the processes involved are so closely allied to classification that it is more convenient to discuss them here.

Any liquid-classifying device can be worked slowly enough to throw down nearly all the solid matter, but the most usual form chosen is the bowl, termed a "thickener" in this field. With no change in essential construction, the bowl with its slowly moving stirrer and central feed can be set to overflow clear liquid, while discharging a thickened sludge of 50–80% solid content in the middle. As already mentioned, when solids of small size are suspended in water they tend to build up some form of solid structure, extending through the liquid phase and only slowly contracting under the influence of gravity. When a mixture of this kind is left undisturbed, it generally separates into four zones. The top layer is clear liquid, and this is followed by a zone of almost constant solid–liquid composition, representing a sort of pseudo-solid column. This crumples at the bottom into a region in which the solid particles, or more probably flocculated masses, are in close contact, with the liquid in the interstices being slowly squeezed out. The final zone is of completely settled pulp, with the flocs as closely packed as is possible under the conditions of experiment.

In any particular problem of separation the rate of growth of the clear liquid region at the top is a measure of the settling capacity of the plant. If this rate is R ft./hr., then $R\rho$ lb. of liquid will separate in each sq. ft. of thickener area per hour, ρ being the density of the clear liquid. When the original pulp contains F lb. of liquid per lb. of solid, and the final sludge D lb., the amount of clear liquid freed per lb. of solid will be $F - D$. It follows that the capacity of the thickener will be $R\rho/(F - D)$ lb. solid/sq. ft./hr. The rate R can be observed in laboratory tests, and will be equal to the *slowest* settling rate observed for any slurry composition between dilution F and dilution D. In a hypothetical settling tank there are initially layers of all concentrations between F and D. These will settle at rates R_1, R_2, R_3 —, with pulp of density ρ_1, ρ_2, ρ_3 — leaving the base of each zone. If all the zones are to remain constant in thickness, then the mass rates $R_1\rho_1$, $R_2\rho_2$ must all be equal. Supposing some variation, it may be supposed that $R_2\rho_2$ is the lowest mass rate; then pulp will enter this zone at a rate $R_1\rho_1$ greater than the rate it is discharged at the base, while below the zone separation to the final separated sludge will be more rapid than the feed entry at a rate $R_2\rho_2$. It follows that the critical zone will expand in volume until it almost fills the thickener, with all other concentrations represented by very thin layers at top and bottom. It is quite common to find that there are two critical settling rates, one with a thin pulp and the other with the final " compacting zone " of settled sludge.

It has been noted that many sludges settle more rapidly when gently agitated. As vigorous agitation would obviously destroy all settling movement, the nature of the gentle stirring is of considerable importance. Generally, the more concentrated slurries are the more affected by agitation, but no general rule applies. In a series of tests on a coal-washery slurry the following figures were obtained for the percentage of solids in the final sludge under various treatments.

	Natural Settling	Settling with Rakes	Settling with Compacting Scraper
Slurry A . .	18	21	28·5
Slurry B . .	42	48	54
Slurry C . .	54	57	64

In the case of a sewage sludge, settling rates varying from 100 with natural settling to 141 with special types of involute spiral stirrer and 130 with ordinary rakes have been recorded.*

Whatever the form of stirring adopted, the final output of any thickener depends upon area and not on depth, so long as this is sufficient for the proper disengaging of the various layers. The thickener must therefore take the form of a large shallow tank with

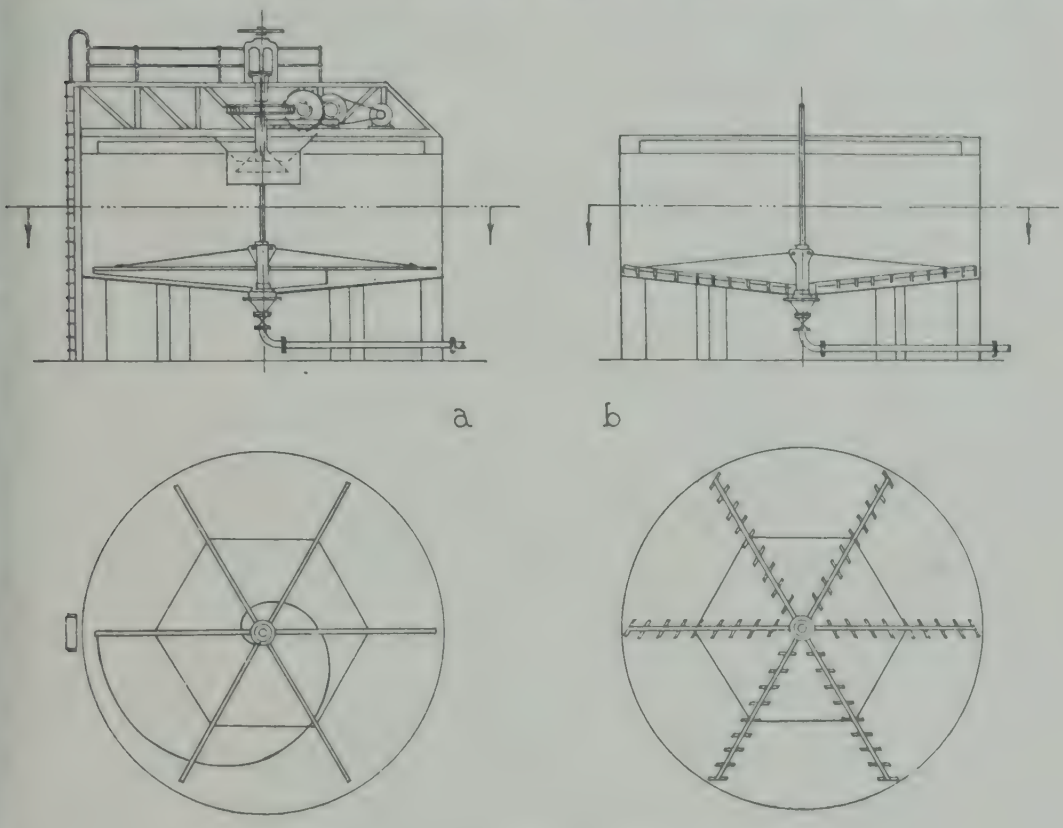


FIG. XIV–14.—BOWL THICKENERS.

a slow-moving stirrer designed to give suitable agitation in the lower layers. Obviously the bowl classifier is eminently suited to this operation, and the rakes can be set to push the settled solids towards the central outlet, just as in classification. The stirring device can take the special form shown in Fig. XIV–14a or the simple rake of Fig. XIV–14b. If a specially thick slurry is required at the bottom outlet, the tanks are made deeper, as this gives a greater degree of compacting of the final zone of settled solids. Where a rapid clarification and a great amount of settling area in a

* Samuel, *Trans. Inst. Chem. Eng.*, 1938, 16, 47.

limited floor area are required, then the shallow bowl tanks can be mounted one above the other in a single casing. The feed enters all the tanks in parallel from central feed points, but the whole tank is filled with liquid. The settled sludge, sliding down a loose-fitting sleeve on the central shaft, forms a seal between compartments, and the clear liquor is drawn off simultaneously from the top of the uppermost bowl and from points immediately underneath the top of the other compartments. The final heavy sludge discharge can be pumped out by a suitable sludge pump, as described in the chapter on Pumping.

The rakes or other forms of stirrer are driven at speeds from 5 to 60 ft./min. at the periphery. This corresponds to one revolution in 9·5 min. up to one revolution in 0·8 min. for a 15-ft. tank. The power requirements will vary with the slurry being handled, but are usually very small. A tank 75 ft. diam., stirred at a rate of one revolution in 10 min., took only 3 h.p. to drive, with a feed containing 5% of solids and a thickened underflow containing 70%.

The old-fashioned settling pond for residual slurries has been replaced by mechanical thickeners, and as a result some very large units have been built. These are stirred by a traction motor, running on a track round the tank edge, and carrying the outer end of a stirrer arm which is pivoted from a central pier. Peripheral speeds are the same as in the smaller models, and the power consumption seems even smaller, with figures as low as 5 h.p. given for a tank 325 ft. diam.

As indicated above, the capacity of any thickening plant is given by the settling rate of the slowest-settling slurry concentration. This can be measured approximately by setting a series of slurries of varying concentration to settle in cylinders in the laboratory. Such tests will give a rate which is lower (as a rule) than that obtained in practice, and will thus form a conservative basis for design. It may be found that the rate of settlement varies inversely with solid concentration, and in this case the capacity of the tank will depend on the concentration of the thickened underflow. This part of the sedimentation cycle may be found to have very different rates of water disengagement from the freer-settling suspensions in the upper regions of the tank. A considerable amount of work has been done upon the problem of slurry treatment to increase (*a*) free settling rates and (*b*) closer packing of the settled solids. It has been found, for instance, that small quantities of starch solution

increase the settling rates of fine coal slurries.* The treatment also appears to decrease the settled volume of the solids, thus limiting the voids and reducing the liquid content. The mechanism of such changes is obscure, and further treatment is outside the scope of the present work.† Clearly the effect of any such addition should be noted in laboratory tests before entering upon design.

An example of the direct application of sedimentation to chemical reaction is found in continuous counter-current decantation, now widely used in the production of caustic soda solutions from sodium carbonate and lime. The reactants—slaked lime suspension and sodium carbonate solution—are stirred for a suitable time (up to three hours) in a series of reaction vessels, and then pass to the first of a series of thickeners. A strong clear solution of NaOH is drawn off, while a heavy sludge of $CaCO_3$, etc., passes on to the next unit. Here it meets dilute solution from the third thickener, and residual NaOH is removed to give a sludge almost free from this product. Washing is completed in a third unit to give a discardable sludge and a wash for the second. The liquor from this second thickener is used to dissolve Na_2CO_3 for the reaction vessels.

Continuous reaction plants of this form are popular because of the steady output and the possibility of labour economy. Where corrosive materials are involved the simplicity of design is an additional advantage. At the same time it is not easy to design a plant into which reactants are fed and from which a steady stream of reaction products will emerge. Often a certain proportion of the feed will "short-circuit" the mixing devices and flow straight through to the product stream.‡ The size of the thickeners required will, as before, depend upon the settling rates of the slurry, but it is generally possible to work to a low solid concentration in the "underflow", and to utilise the more rapid settling rates obtained in dilute slurries. As an example of the use of laboratory data an example based on "causticising" is given below.

Example. *A reaction slurry from* $Ca(OH)_2$–Na_2CO_3 *contains* 10% *of solid. It is proposed to treat this in three thickeners, removing a clear overflow and a sludge containing* 30% *of solids. The slurry is to enter the settling system at a rate of* 10,000 *lb./hr., and the liquor density will vary from* 66 *lb./cu. ft. down to* 62·5 *lb./cu. ft.*

* Wilkins, *Trans. Inst. Chem. Eng.*, 1938, 16, 54.

† Donald, *ibid.*, 1940, 18, 24.

‡ See, e.g., Denbigh, *Trans. Farad. Soc.*, 1944, 40, 352.

The amount of liquor to be disengaged in the first settler will be 6660 lb. or 107 cu. ft. A series of laboratory tests with the slurry gave the following results:

Slurry Concn. (% solids)	Rate of Settling (in./min.)	Rate of Liquor Disengagement (cu. ft./sq. ft./hr.)
10	2	10
20	1	5·0
30	0·5	2·5
40	0·1	0·5
50	0·02	0·1

As a 30% underflow is required, the area of the thickener must be 107/2·5 or 43 sq. ft. The bowl must be 7 ft. 6 in. diam. The slurry depth is a matter for experience, but should not be less than 5 ft. to allow for incidental disturbances. Further laboratory tests on the slurry diluted with water will give the size of the succeeding thickeners.

The overriding importance of settling rates is clear from the above example. If the underflow had been "thickened" up to 40% of solids the size of thickener would have been increased five-fold. When very large amounts of liquid are being handled, the importance of a very slight change in settling velocity is obvious. It is general to express these settling rates in terms of area per ton of thickened slurry solids per day. Thus, in causticising plant separating lime mud with 40% of solid, the area required ranges from 8 to 20 sq. ft./ton/day. Flotation concentrates of heavy metal ores usually require 3–10 sq. ft., while blast-furnace dust from a washer, entering as a very dilute suspension, may need as much as 240 sq. ft./ton/day to thicken to 40% solids underflow.

In addition to the normal stirred gravitational thickeners, certain forms of filter-thickeners have been developed, in which a filtering device is used to give a clear filtrate and a thickened slurry. In the Oliver-Borden thickener a number of filter tubes—steel frames covered by a filter cloth—are suspended in a tank with a vee-shaped base. The tank is filled with the slurry to be thickened, the tubes put under suction as in normal filtration work, and a solid cake built up to a suitable thickness. Suction is then cut off and an air-blow detaches the cake, which falls to a repulping stirrer in the vee-base of the tank. A conveying device combined with the repulper can clear the thickened slurry out of the residual liquors without interrupting the operation of the plant. The thickened sludge may contain up to 70% of solids, and outputs of 100–500 tons of solids per

1000 sq. ft. of filter area can be obtained per day. As it is possible to pack this amount of filter area into a tank of perhaps 150 sq. ft. in area, a considerable increase in output over normal thickeners of the same floor area is obtained. At the same time the slurries giving this high throughput may well be handled in ordinary filter units, and clearly the extra cost of vacuum and air-blowing militates against the use of this form of plant.

Another form of filter-thickener utilises gravity settling to remove

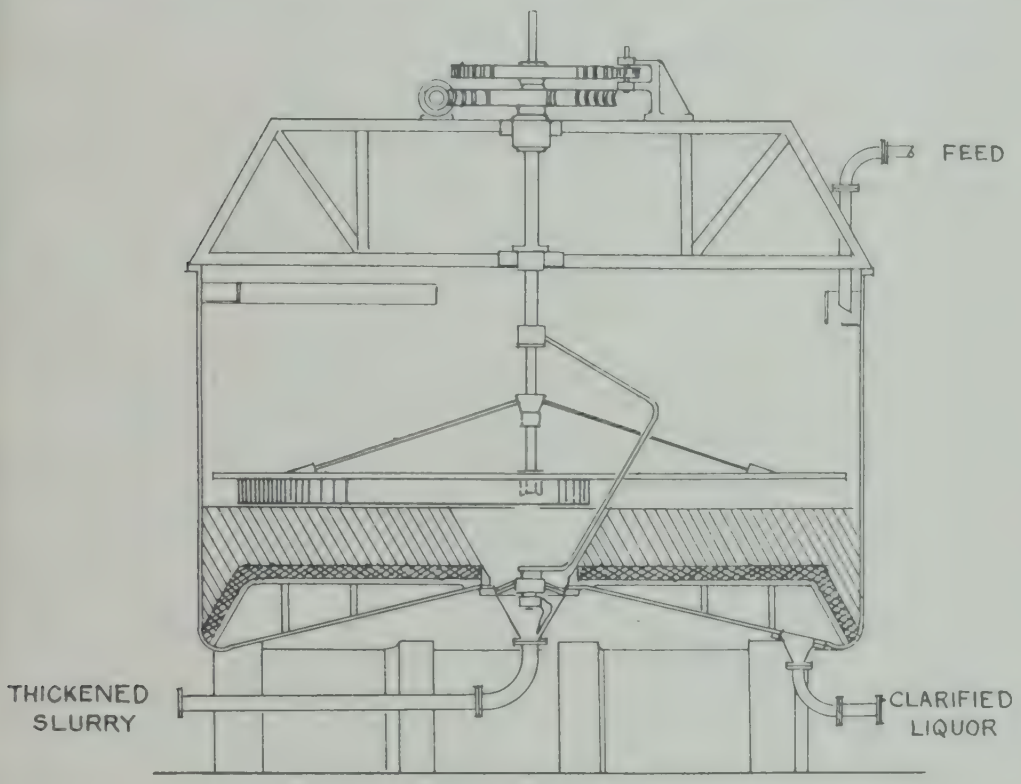


Fig. XIV–15.—Filter Thickener.
(*Courtesy of International Combustion, Ltd.*)

solids and at the same time to give a clear filtrate through a sand bed. The design is shown in Fig. XIV–15; the slurry entering at A settles steadily on the sand bed at B, and is raked to the central discharge by the spiral scraper. A thin layer of sand is also removed, giving a fresh surface for filtration of the liquid, which passes away as a clear filtrate at C. The sand bed is removed at rates which may be as low as $\frac{1}{64}$ in. per day, and as the original bed is some 5 ft. in thickness, long periods of steady work are obtained before the bed of sand needs renewing.

CHAPTER XV

SOLID SEPARATION

Solid materials may be separated by means of differences in density, shape, magnetic or electrical susceptibility, and surface characteristics. These properties vary very widely, and no one general method applies to all cases. The various properties may interfere with each other, a shape factor checking separation by density, and surface characteristics masking electrical properties. A very large number of separation processes have been devised, perhaps more for ore refining and coal cleaning than for what are commonly understood as chemical processes.

The first and simplest of these operations is that of separation by density. The rate of fall of a solid particle in a fluid has been shown to be proportional to the effective density $S - \rho$ where S is the true density of the solid and ρ the density of the fluid. If two solids have the same size and shape characteristics but different densities, it is possible to use the methods of size separation by classification to separate the two. The process is made less easy by the fact that few solid materials break in the same way, and in consequence the falling velocities may be greatly modified. A process for separating mica from granitic masses may be given as an instance. When the mica-bearing stone is broken down to 10-mesh fragments, the mica forms flakes and the other constituents rounded lumps. The materials all have approximately the same density—if anything the mica is slightly heavier—but an upward-current classifier floated off all the mica flakes, together with the smaller rock fragments. The floats passed to a 20-mesh screen, which passed all the small rounded particles, leaving a pure mica concentrate.

When the solids to be separated have less obvious differences in shape, it is still necessary to crush to a very even size if upward-current classification is to be used. This has militated against a wide application of the principle, and upward-current density separation plants are rare. Perhaps the best known is the Rheolaveur coal washer, designed for the separation of shale of 2·6

density from coal of 1·4 (C.G.S. units). The Rheolaveur is a long narrow trough (Fig. XV-1) down which flows a slurry of water, coal, and shale. The solids have been crushed to a close size range such as $\frac{1}{2}$ in. to $\frac{1}{4}$ in., and a separate trough is used for each size range. At intervals in the trough base are slit openings (pockets) up from which a water current flows at a rate sufficient to float off the coal, while allowing the shale to fall through into the bottom of the pocket. The valves at the base of these pockets, as shown in Fig. XV-1, are opened at intervals to discharge a thick sludge,

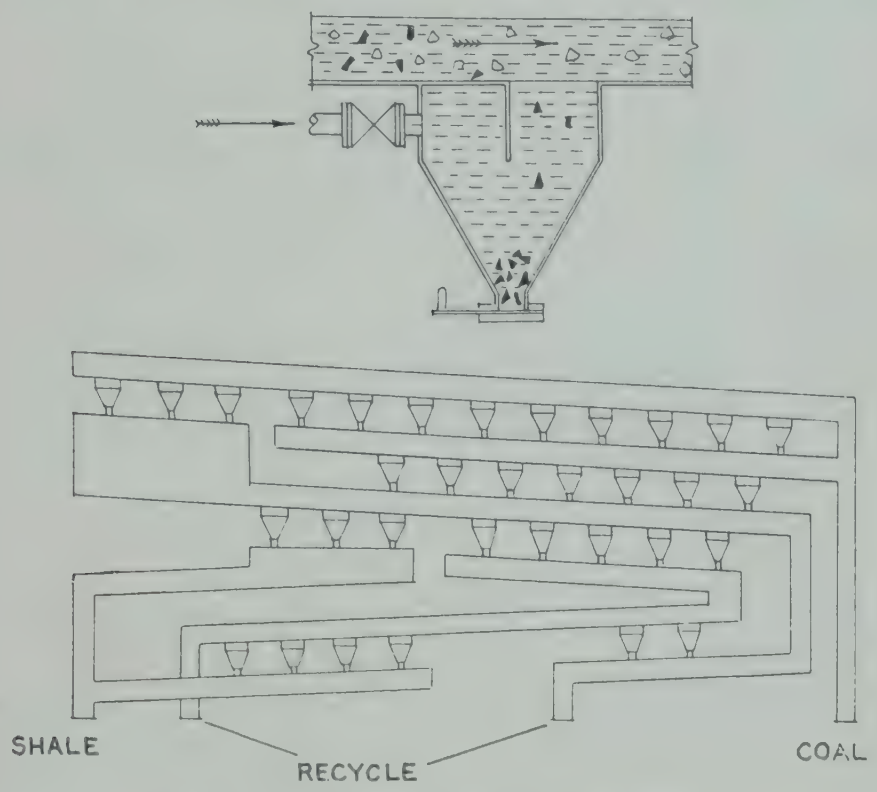


Fig. XV–1.—Rheolaveur Coal Washer.

while the cleaned coal flows to screens at the base of the trough. The separation is aided by the fact that the slurry tends to form a pseudo-fluid of density equal to the mean of the constituents. Then the lighter coal will float and the heavier shale will sink—an indication of the "heavy liquid" processes which are now being widely adopted.

The same fluid formation is even more marked in the case of jig washers, which have been very widely used for all forms of solid separation. In a typical washer of this form (Fig. XV-2) a stream of solid-liquid mixture passes down one side of a U-shaped trough

with a central partition and a supporting screen for the solids in the slurry. On the other side of the central partition a plunger or a pulsating air-flow set up a regular up-and-down motion—" teetering "—of the solid liquid slurry. This keeps the whole of the solid in a " fluidised " state, and relative movements bring the heavier fragments to the bottom of the slurry bed. The precise mode of operation of the jig is still obscure.

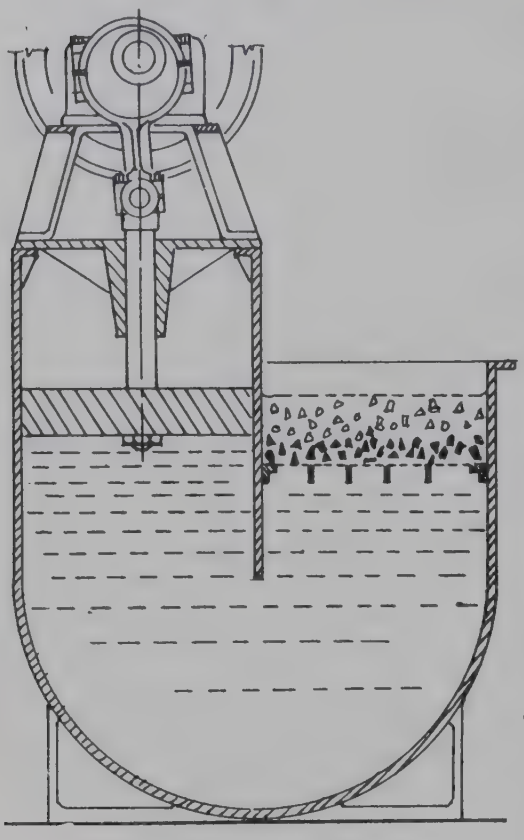


Fig. XV–2.—Jig Washer.

When a particle is just starting to fall in a stationary fluid, the weight acting to cause movement is $K_1 d^3(S - \rho)$. There is no force-resisting motion, as $V = 0$ and the weight acts on a mass $K_2 d^3 . S/g$. The initial acceleration is

$$\frac{K_1 d^3(S - \rho)}{K_2 d^3 . S/g} \text{ or } K . \frac{S - \rho}{S} . g$$

Heavier fragments will therefore start to fall more rapidly than lighter ones, irrespective of relative size. This action is not so important as the rapid upward surge of the slurry at the downstroke of the plunger, while the entraining effects of shape also play a great part. It seems that jigging effects are as selective as the mechanism of flotation, and this has found expression in the number of designs and methods of working of this form of washer.

Whatever the mechanism, as the slurry moves along the length of the washer it is stratified into a heavy lower layer and a light upper layer. Although it is not necessary to start with a very definite size range, it has been found in practice that best results are obtained when the largest fragments are not more than twice the size of the smallest, while it is not practicable to jig-wash lumps of more than 3 in. in size. Separation between the two layers is usually made by a submerged weir, under which the heavy material flows, while the lighter fragments float over the weir top. The power requirements

are not small. A unit 8 ft. long and 2 ft. 6 in. wide took 20 h.p. in cleaning 7–10 tons/hr. of small coal. This represents an output of some 1000 lb./sq. ft./hr.—a high figure. Where the concentrate is the heavier fraction, the figure is from 300 to 600 lb., but power requirements are lower, only 5 h.p. being required for four 3-ft. square cells, in series, dealing with low-grade sulphide ores. An example taken at random from jigging practice shows a feed containing 4·1% of lead, from which a concentrate containing 70·0% lead could be separated.

An alternative to jig-washing is the use of a shaking table, which

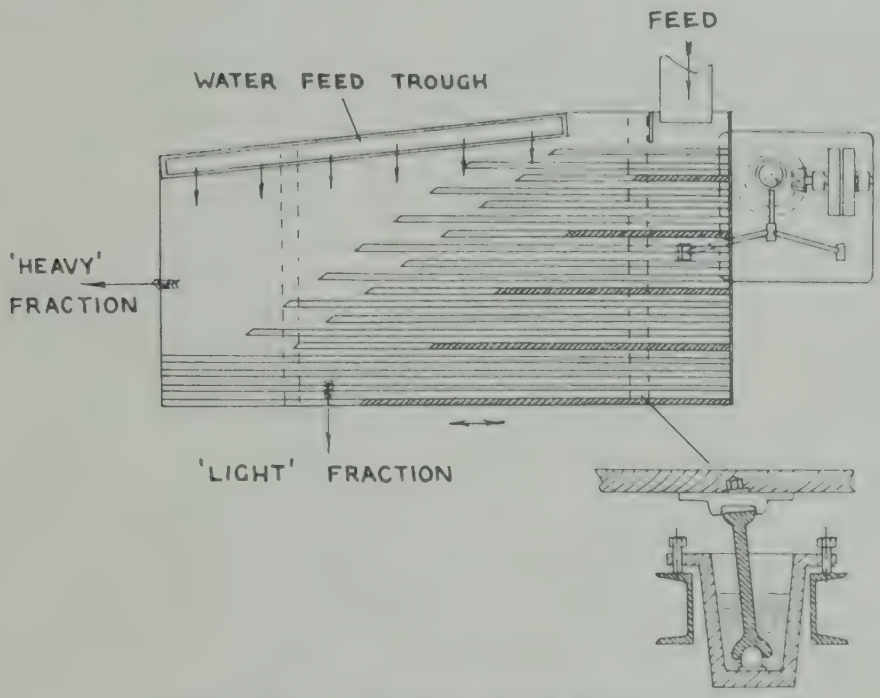


Fig. XV-3.—Shaking Table Separator.

is a corrugated surface, usually about 18 ft. by 6 ft. in size, though wide variations are possible. The surface is set at a slight slope down from the feed side, so that a slurry feed tends to flow straight across the table, at the side which is (Fig. XV 3) most fully baffled by "riffles". These riffles are from $\frac{3}{4}$ in. to $\frac{1}{8}$ in. deep and are made in varied shapes. The heavy fragments are trapped in the riffles by the natural flow of the slurry and by the shaking of the table, which jolts from side to side along the axis of the table at right angles to the feed flow. Tables of this kind are only used for fines of below about 10-mesh, and can be used on dry powders, with air blown through perforations on the table to give fluidity to the

solid bed. In wet working a table 15 ft. long and 6 ft. wide can deal with 2·5 tons/hr. of a mixture containing 5% of sulphide, giving a concentrate of 60% content, and taking some 0·5 h.p. for driving at 255 r.p.m. In the dry cleaning of coal a table of the same size had an output of 6 tons/hr. of coal, with a power consumption of 1–2 h.p. Again, as with jig-washing, a large number of modifications of the basic plant shown in Fig. XV–3 have been devised and operated. Details of these and their operating characteristics will be found in books on ore dressing and metallurgical practice.

There appears to be a growing tendency to replace other forms of gravity separators by " heavy liquid " processes. These are based on the simplest of all principles, in that a fluid is made up in which the heavier components will sink, while the lighter components will float, irrespective of the size or shape of the fragments. In the case of coal cleaning, where coal of a density of 1·2 to 1·5 was to be separated from shale of about 2·5 density (water = 1), the provision of a heavy liquid was not too difficult. Zinc and calcium chloride solutions can be made up to a density sufficient to float the coal, while mixtures of carbon tetrachloride and benzene will give a wide range of separating fluids. Unfortunately, these solutions are either costly, corrosive, or toxic and in general processes based on them have not attained commercial success. When the solids to be separated were all above 2·5 in density, and the heavier fractions were mineral sulphides of up to 7·5, such liquids as the above were much too light. They have now been replaced, with considerable commercial success, by suspensions of finely divided solids, such as sand (density up to 1·5), clay (up to 1·4), magnetite (up to 2·5), galena (up to 3·3), and ferro-silicon (up to 3·2). Lead powder will give a suspension of up to 5·0 density, but the suspension is rapidly oxidised and is too reactive for practical use.

The typical heavy-liquid flotation unit is extremely simple in operation, as shown in Fig. XV–4. The solid feed, which has no size restriction, and may be up to 12 in., or run of mine product, passes down a chute into the separating bath, which is pyramidal in form. A series of surface rakes break up the " floats " to ensure that no heavy fragments are trapped thereon, and move the mass forward and over a weir to separating screens. A water-spray washes off the residual separating fluid, which is carried down to further screening and remaking devices. Meanwhile at the base of the primary separator a slow upward current raises any medium-

density fragments, or "middlings", through a rising limb to another screen, while the heavy residues are lifted from the bottom of the pyramid by a conveyor. The fines of the feed are inevitably entangled with the suspension, but by bleeding off a portion of this to a subsidiary separating device, such as a flotation cell or magnetic separator, it is possible to regenerate the suspension for further treatment.

In a plant as shown in Fig. XV–4 coal of 12 in. down to 2·25 in. could be separated into 86·4% clean coal of which 100% floated at

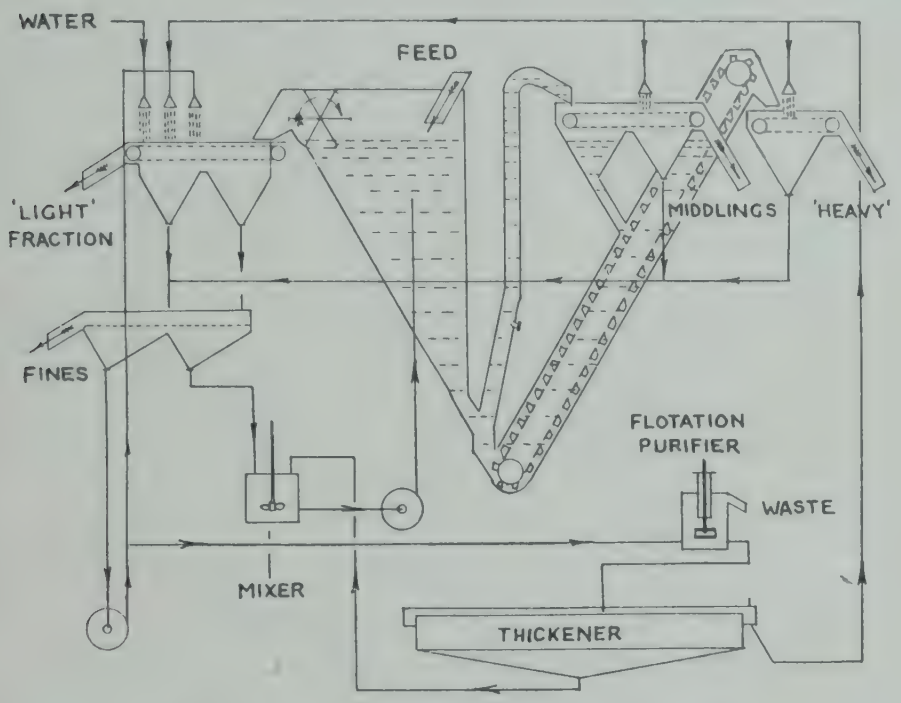


Fig. XV–4.—Heavy-Liquid Flotation Unit.

1·44, and a shale residue (13·6%) of which 99% sank in fluid of 1·40. An almost theoretical separation has been made between coal substance and mineral contaminant. Results for smaller coal were not quite as good, owing to the formation of a middlings fraction. In ore separation a feed containing 6·3% Pb and 3·5% Zn is concentrated up to 12·4% and 6·5% respectively, with recoveries of over 90% in each case. A separator 8 ft. wide by 9 ft. deep can deal with about 70 tons/hr. of a typical feed mixture, and the total power consumption is of the order of 1 h.p. hr. per ton of material treated.

An interesting development from this is the use of dense liquids

to secure a separation of very clean coal from ash residues. It is generally true that large solid fragments, of above $\frac{1}{4}$ in. size range, are best suited to float-and-sink separation, as they can easily be separated from the suspensions with a solid size of less than 100-mesh which are used for separation. With coal such pieces are still liable to contain ash inclusions, and it is necessary to crush this to about 1 mm. size (18-mesh) to expose these for separation. The dedusted coal is then cleaned in a long narrow bath containing a suspension of magnetite. A coal containing initially 13–14% of ash was separated into a fraction (76%) containing 1·5–2%, a middling (8%) with 25%, and a shale residue (16%) with 65% ash. The products are sprayed with water to wash off the magnetite slurry, and this "spent" reagent is passed to recovery tanks. In these tanks a magnetised wheel carries a belt conveyor section; as the wheel rotates, the belt carries away a cake of magnetite particles in a manner reminiscent of a rotary vacuum filter with a string discharge. These particles are redispersed for further use. One of the great advantages of magnetite suspensions is the ease with which the slurry may be purified and reused, and by combination with ferro-silicon (15% Si), which shares this advantage of magnetic property, the most popular forms of "heavy liquid" can be built up.

The process of revivification can be taken as an example of the application of magnetic susceptibility to separation. These processes are seldom used in chemical plant.*

Flotation processes are much more important, and are still extending in scope. They rely on the relative ease with which solid materials are wetted by liquids. As a rule metallic sulphides resist water wetting, while clays, quartz, and complex earths are readily wetted. This separation is of such importance in the metallurgical industries that the practice of flotation was largely built up on sulphide flotation. The process is most easily put on a quantitative basis by study of the "wetting angle" θ. In case A of Fig. XV–5 a drop of water on the solid surface retains its spherical form almost completely, while if the solid is submerged in water an air bubble spreads out flat on the surface. The constancy of the angle θ will be noted, and the close approach of this angle to 180° represents poor wetting and a solid that can be floated. If the angle θ is less, indicating a greater ease of wetting, then the water drop is flattened, but

* But see, e.g., Wilkins, *Trans. Inst. Chem. Eng.*, 1946, 24, 6.

the air bubble is not so firmly attached to the surface. Finally, with very easily wetted solids, θ approaches zero, the water spreads easily on the solid, and the air bubble is attached by a tiny point of contact. These two latter are set out in B and C of Fig. XV-5.

The wetting angle is a specific characteristic of the solid-liquid interface, generally somewhat modified by hysteresis effects. When a mixture of two solid materials, one of which has a high wetting angle with water, is beaten up with water and air to form a foam, the air bubbles attaching to the solid, as in Fig. XV-5 (A), will raise these particles to the surface. If only air and water are used the effect is very transitory, and the foam breaks up immediately

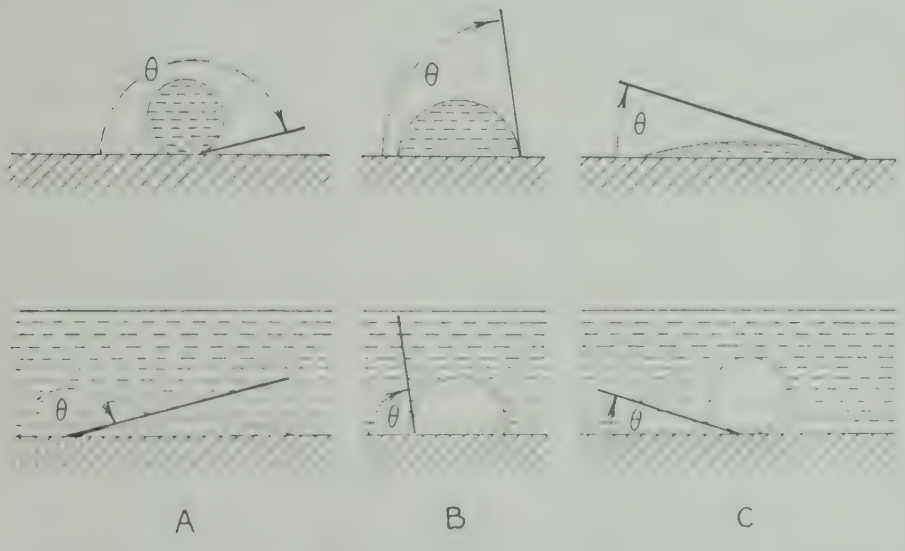


Fig. XV-5.—Wetting Angles.

stirring ceases. A frothing agent is therefore necessary which will stabilise the foam to a degree sufficient for it to be removed from the flotation cell. This froth stabiliser may also act as a promoter of froth formation by lowering the aqueous surface tension. This is not strictly necessary, but the most usual frothing agents do lower the surface tension of the water to some degree.

Pine oil, creosote oil, mineral oils, oleic acid, and some of the higher alcohols have all been used as frothing agents. The amount required is small, from 0·1 to 0·5 lb./ton of ore treated, but the frother is now generally modified in its action by other reagents. These are known generally as "collectors", "activators", and "depressants", without any very rigid specification. The most popular collectors, evolved originally to deal with sulphide-containing

mixtures, are the salts of xanthic acid, such as sodium ethyl xanthate—$S=C\begin{matrix}O\cdot C_2H_5\\ S\cdot Na\end{matrix}$. These are particularly suited to the lifting of sulphides, and are supposed to form a water-repelling film on the ore surface, with subsequent alteration of the contact angle. It may be noted here that the wetting angle for sulphide ores treated in this way is about 60°–90°, while the angle for clays, etc., may be about 5°. The amount of collector is usually small, as an excess above about 0·3 lb./ton often reverses the original action, perhaps by forming a second molecular layer. A number of these collectors have been devised, many of very considerable chemical complexity. For metallic sulphides the collector is usually one with a xanthate structure, but when other materials have to be " parted " the range of composition is wider. Ordinary soap, in a weak alkaline suspension, can collect silica from alumina, while a substituted thiourea can float talc from siliceous residues.

The action of collectors can be modified by " depressing " or " activating " agents. These make the action of the collectors more selective by reaction with one or more of the constituents of a complex mixture. The *p*H of a solution is the most common operator in this way, and a move in either direction from a neutral point can bring about decrease in wetting angle to a greater or less degree according to the nature of the surfaces presented. More specific agents can be found, such as sodium cyanide for the " depressing " of zinc when lead is being floated. Sodium sulphide acts as an activator in the flotation of metallic oxides, perhaps through a partial reduction of the solid surface, and a number of other agents have been used.

In a typical case of selective flotation an ore containing 15% of both lead and zinc sulphides was given two treatments in series. In the first the frother was eucalyptus oil, the collector ethyl xanthate, and the depressing agent (for the zinc sulphide) sodium silicate. The floats were lead sulphide of 80% purity, containing over 90% of the original lead. In the second treatment, with the frother of eucalyptus oil as before, the collector was sodium aerofloat,

$$S=P\begin{matrix}O\cdot C_2H_5\\ O\cdot C_2H_5\\ S\cdot Na\end{matrix}$$

with copper sulphate as an additional collector or activator for zinc,

and lime as a depresser for iron sulphides. The yield of zinc sulphide was very much the same as for the lead.

Some remarkable separations have been claimed for flotation, perhaps the most striking being the separation of KCl crystals from NaCl. The flotation mixture was sodium aerofloat and oleic acid, which worked on a suspension of the mixed crystals in a saturated solution to give a froth of KCl suspension. The main difficulty in flotation is that all the material must be ground to below 60-mesh, and preferably to the 100–200-mesh range, while avoiding fine slimes. However, this size range is being extended upwards, and

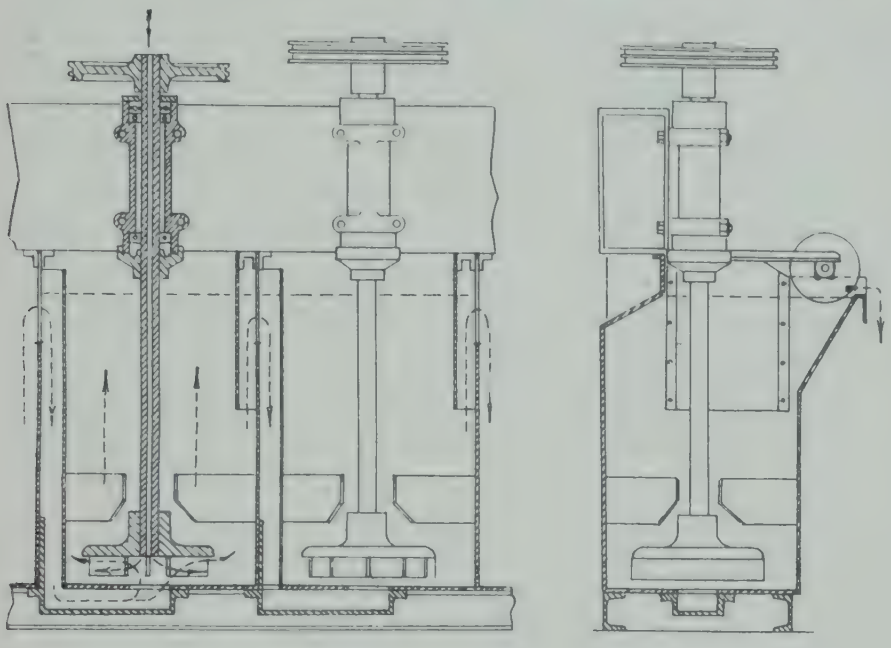


Fig. XV–6.—Flotation Cell.

special froth-strengthening agents allow for operation, in coal cleaning, at sizes up to 0·25 in.

An aeration cell must be designed to give intimate contact between air, water, and the finely divided solid, which is usually pumped to the flotation plant as a slurry of 10–25% solids. One of the simplest ways in which this can be done is to mix the slurry at high speeds with a turbo-mixer fitted with a hollow shaft or an air lead baffle, as shown in Fig. XV–6. The feed enters from the side of the cell, a number of which are generally used in series, and is dispersed in the body of the cell with air drawn down the central shaft. The foam is swept from the top by a froth-removal paddle, while the residual solid slurry flows out on the far side of the cell. These cells are

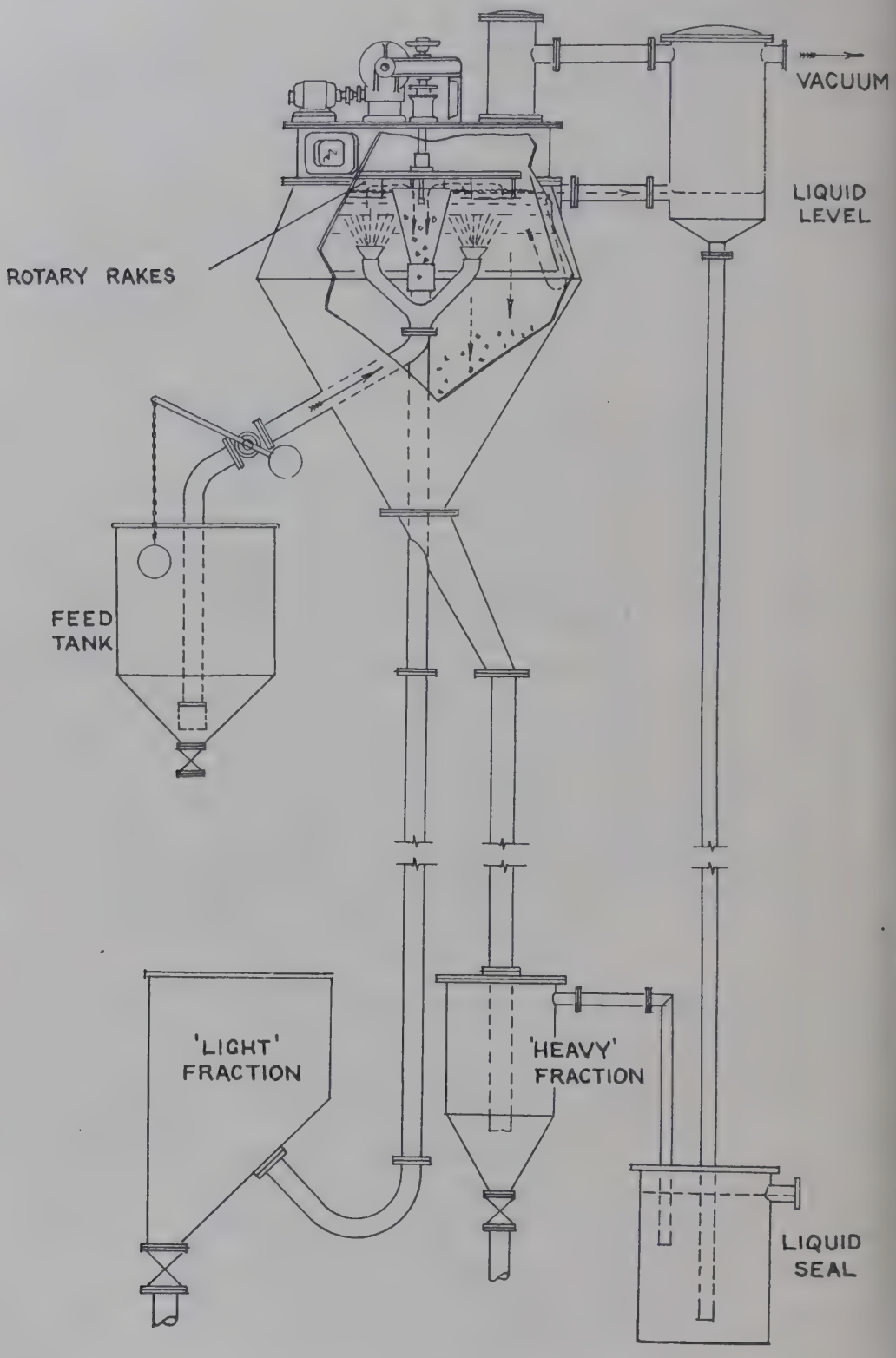


FIG. XV–7.—VACUUM FLOTATION.

built in sizes from 2 ft. square up to 4 ft. square, and a 6-cell battery of the largest size will process 1000 tons per day of solids, used as a 20% suspension in water. The power consumption is about 10 h.p. per cell, or 60 h.p. in all (some 1·4 h.p. hr. per ton). The cell shown is one of a large number of possible modifications, known as "sub-aeration" machines. Other forms utilise an air-lift process to aerate and agitate the slurry, or blow air through the perforated base of a slurry tank.

An altogether different design is used in the Elmore vacuum flotation system. This is principally used for coal cleaning. As shown in Fig. XV 7, the fine coal (all below $\frac{1}{8}$ in.) is mixed with reagents and water and flows into a rising main of gradually increasing cross-section, terminating in a large cone-shaped head maintained at 27 in. of vacuum. The froth of clean coal which is formed overflows a circular launder and returns to the foot of the plant, the froth collapsing as it falls owing to the increased pressure. The refuse falls to a side outlet for intermittent discharge. The capacity of a 5-ft. diameter cone is from 25–50 tons per day of solid feed, with a total power consumption of 5 h.p./hr.

The foregoing paragraphs have covered very incompletely the technique of a very important branch of metallurgical technology which is rapidly expanding its scope. The development of processes for the concentration of radioactive ores, where the very valuable nature of the concentrate allows of expensive processing, is typical of these developments.* In the British Isles the principal applications of flotation have been in the field of coal cleaning, and the floating of sulphide ores has been mainly carried out in Australia, America, and South Africa. Most developments have therefore been reported from these centres, but with the growing need for working low-grade ores this may not always be the case.

* McTaggart, *Trans. Inst. Chem. Eng.*, 1942, 20, 65.

CHAPTER XVI

MEASUREMENT

It is vital in all large industrial units to know exactly where all materials are going. A complete mass balance of everything entering a process should always be drawn up, as fractions of 1% of a chemical yield may accumulate in unsuspected corners and appear with unpleasant results after a certain lapse of time. Any mass balance entails precise measurement and it is equally important to measure rates of flow for process control inside the plant. Ideally all materials should be weighed, as the most accurate way of measurement, but this is very often impracticable. Direct measurements of volume in a static tank may replace weighing for liquids and are not unusual for gases, but it is often necessary to rely on an integrated rate of flow.

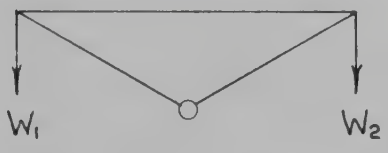


Fig. XVI–1.—Weighing Principle.

Weighing-machines in industrial practice are usually of the steel-yard type and range from the small platform scale of 3 cwt. capacity to the large weighbridge weighing up to 50 tons. These can be obtained with direct-reading dials or with steelyards for hand operation. The latter are more shockproof and less liable to get out of order with rough usage. Where a steady supply of weighed charges is required, a balance system built up on the tilted arm principle is used. As will be seen from Fig. XVI–1, a balance arm of V form, poised around the tip of the V, is inherently unstable; as soon as the weight W_2 exceeds W_1 by a slight amount, the arm tips, leverage of W_2 increases as the leverage of W_1 decreases and the arm moves over to the right with sufficient force to open or close a valve. The discharge from a bin of solids can conveniently be measured and recorded in this way, as can also the flow of a liquid, such as water flowing to a boiler. The tilt of a tipping bucket can be used to measure the water and feed appropriate amounts of water-conditioning agents at the same time.

An alternative method of providing fixed weights of solid in a given time is to feed by a weighbridge conveyor as shown in Fig. XVI–2. A section of the conveyor belt is supported on counterpoised rollers, which move a gate on the discharge from the solid hopper and adjust the mass on the belt to a fixed figure. It will be noted that the height of the solid stream on the conveyor belt is altered; this setting of the height of the ribbon discharged from a valve on to a moving surface is a principle widely used in handling large quantities of solid. A belt conveyor can act as a measuring device for several solid bins discharging to give a solid mixture. The ribbons from each successive bin overlay each other and the whole stream is discharged into a mixing bin. A unit of this type has been used

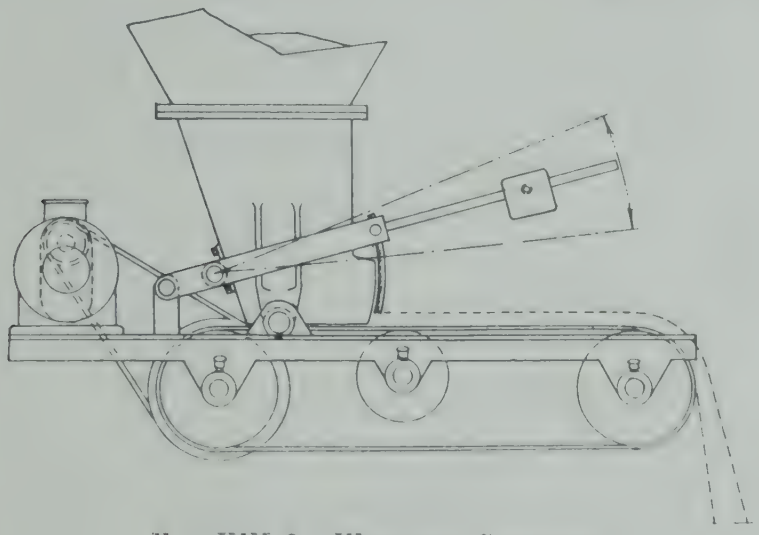


Fig. XVI–2.—Weighing Conveyor.

for blending small coals at a total rate of 100 tons/hr. for coke-oven work. It is not very accurate and a series of rotary feeders, as shown in Fig. XVI–3, is more popular for this large-scale blending. Each of these feeders discharges on to a common conveyor belt, and this into a bin, commonly the boot of a bucket elevator. The feeder is a rotating plate, which is either mounted below the outlet of a solid hopper or forms the base of a solid bin (this for smaller quantities). A ribbon is peeled off from the bottom of the solid cone formed at the base of the hopper by an adjustable knife and discharges over the edge of the plate. It will be realised that this, as with the use of conveyor ribbons, is a measurement of solids by volume, but where composition of solid from each bin is constant, the final mix does not vary much. In the mixing of components for

a cement kiln, at a rate of 5 tons/hr., the content of any one of five components did not vary by more than $\pm$ 1%.

It should be remembered, with all handling of granular solids, that although these substances flow, they are not liquids. In particular, the rate of discharge from an opening at the bottom of a tank is independent of the "fluid" head and is determined by such factors as particle size relative to size of opening and surface roughness of individual particles. These abnormalities are also present to some extent with liquid–solid suspensions, which always tend to exhibit thixotropic properties, such as sharp variations in viscosity with rate of shear. The steady flow of granular materials through a small aperture can be used for measuring, but the solid will always tend to "bridge" over the hole.

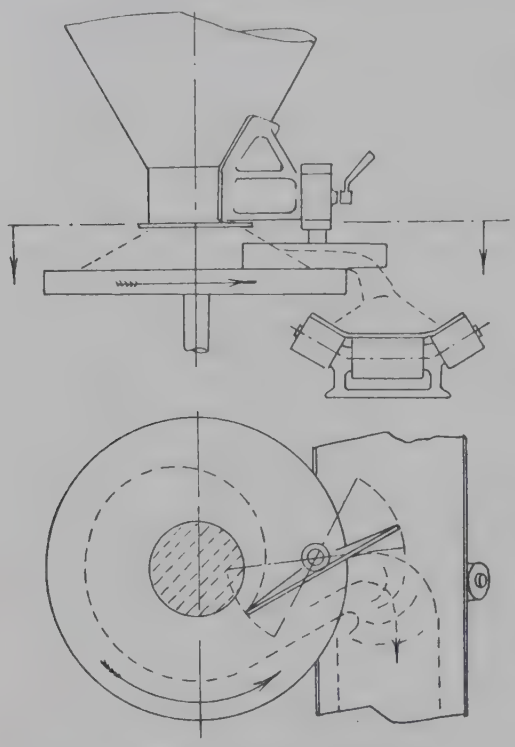


Fig. XVI–3.—Rotary Feeder.

The rate of flow of true fluids, either liquids or gases, is more easily measured. In the first place, if steady flow is established in any suitable section of a chemical plant, the pressure difference between inlet and outlet may be used as a guide to working. Any long straight length of circular pipe, for instance, will cause a drop in pressure. This is determined by the fundamental formula

$$P = 8\left(\frac{R}{\rho v^2}\right) \cdot \frac{L}{D} \cdot \frac{\rho v^2}{2g}$$

Here P is the pressure drop in lb./sq. ft. for a pipe of length L and diam. D in which fluid of density ρ is flowing with velocity v, all figures being in F.P.S. units (p. 4). It is seldom that such a convenient conduit is found and recourse to some specific device is usually necessary. A carefully kept log of pressure drops in a plant is, however, of considerable use in detecting irregularities before any serious incident occurs. For instance, a continuous-pipe still was worked at

constant liquid feed until an initial pressure drop of 20 lb./sq. in. had increased (over 200 days) to a pressure drop of 40 lb./sq. in., when the still was shut down for cleaning.

The most usual method of estimating fluid flow is by pressure drop across some type of orifice. At this "orifice", or constriction in the flow channel, static pressure is, in an ideal fluid, converted into velocity. It follows from fundamental considerations that the difference in pressure between fluid in the orifice and in the main stream before acceleration has occurred will be proportional to the square of the change in velocity. In general terms $v_1^2 - v_0^2 = 2gh$, where v_1 and v_0 are velocities in the orifice and conduit respectively and h is the difference in fluid head. All fluids, whether liquid or gaseous, show some divergence from this ideal expression. Gases change in volume and temperature, while both liquids and gases

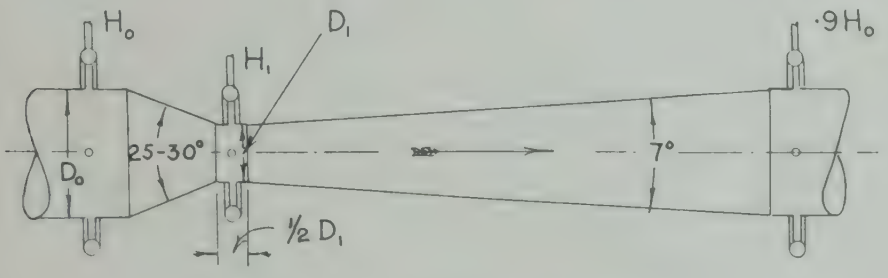


Fig. XVI–4.—Venturi Tube.

give flow patterns through the orifice which make it difficult to estimate the true value for h. In these circumstances it is necessary to use some standardised constriction form and to use various correction formulae which have been experimentally established.

The most accurate form of orifice meter is the Venturi tube, shown in Fig. XVI-4. This has an upstream entrance matching the conduit in diameter D_0 followed by a convergent cone frustrum with a vertex angle of 25°-30°. This is followed by a cylindrical throat section of diameter D_1 (from $\frac{1}{2}$ to $\frac{1}{4}$ D_0) and length not more than $\frac{1}{2}D_1$. The downstream cone frustum is long and narrow, with a vertex angle of about 7°. The main function of this tailpiece is to promote the conversion of velocity to fluid head with the minimum of energy dissipation. Pressures are taken from ring tappings just above the entry cone and at the throat, care being taken to keep the inner surfaces of the channel as smooth as possible. In the diagram

of Fig. XVI–4, if H_0 is the fluid head at the entry, and H_1 the head at the throat, then

$$(H_0 - H_1) = \frac{v_1^2 - v_0^2}{2g}$$

If the fluid is relatively incompressible, and the ratio $(D_1/D_0)^2$ is x, then $v_0 = xv_1$ and $2g(H_0 - H_1) = v_1^2(1 - x^2)$

$$\text{or } v_1 = \sqrt{\left[\frac{2g(H_0 - H_1)}{1 - x^2}\right]} \quad . \quad . \quad . \quad . \quad . \quad (1)$$

No matter how carefully the Venturi tube is made, slight local disturbances and surface drag make the effective throat diameter less than D_1, while some of the pressure drop must be due to fluid friction. These effects are covered by a correction factor C, so that

$$v_1 = C\sqrt{\left[\frac{2g(H_0 - H_1)}{1 - x^2}\right]} \quad . \quad . \quad . \quad . \quad . \quad (2)$$

The value of C has been shown to vary with the degree of turbulence in the throat section. When the value of Reynolds' Criterion at this point exceeds 10,000, then C for a well-made tube approximates to 0·98; it drops to 0·95 for a Reynolds value of 1000, and then falls off rapidly as flow grows slower. A Venturi tube should be chosen of such a size that the flow being measured is always well inside the turbulent region. When this precaution is taken, the use of equation (2) will give results correct to $\pm$ 1% for all liquids, and almost equal accuracy for gases if the pressure drop is small compared to the total pressure. Where larger pressure drops are involved (above 6 in. w.g. for gas at atmospheric pressure) a number of corrected formulae have been suggested, but recourse should be made to specialised publications for these.* Despite the long re-entry cone of the Venturi, only about 90% of the velocity head is converted back into static pressure. The length of the meter makes it unwieldy and costly, while it can seldom be inserted into an existing pipe-line. For these reasons the meter is seldom used in chemical plant, the less accurate but much more convenient orifice or nozzle plates being used instead.

The differential pressure $(H_0 - H_1)$ is hardly ever read directly in terms of the fluid passing the meter. It is usual to employ in the gauges a liquid that can be clearly seen, and to measure the

* See, e.g., B.S. 1042, 1943, or A.S.M.E. Res. Publ. " Fluid Meters ", 1937.

pressure differential H_w of this. Then if the fluid in the conduit has a mean density ρ_1 and the gauge liquid a density ρ_2, it follows that

$$(H_0 - H_1) = H_w \cdot \rho_2/\rho_1$$

In most cases a simple U-tube gauge is connected between the tapping points, with an air seal between the conduit connections and the liquid in the gauge. If the pressure difference is small, then the U-gauge can be tilted, as in Fig. XVI–5a. The normal limits of accuracy in reading a vertical gauge are $\pm$ 1 mm. If the gauge is tilted to a slope of 1 in 20, which represents a practical limit,

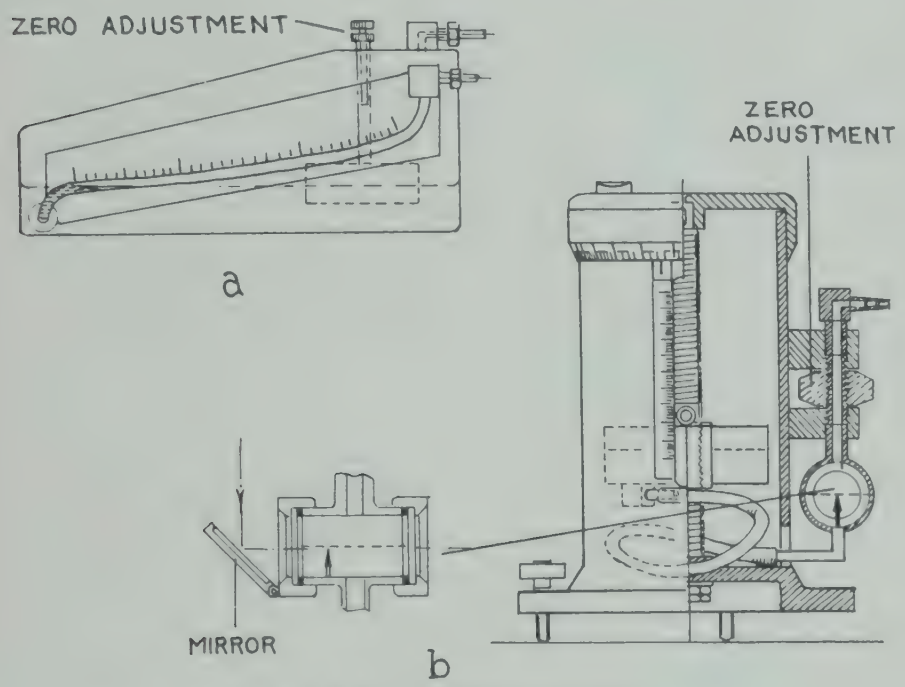


Fig. XVI–5.—Micro-Gauges.

then a similar variation gives an accuracy of $\pm$ 0·05 mm. of fluid head. Smaller pressure differences than this must be read on some form of micro-gauge, such as that illustrated in Fig. XVI–5b. Here a reservoir moves on a micrometer screw to bring the water level in the outer reservoir back to a standard position. The micrometer head can be read easily to 0·01 mm., but it is difficult in practice to secure results nearer than 0·02 mm., owing to optical errors. It is seldom that such low pressures are encountered with Venturi tubes or orifice plates, but they are the usual condition for measurement of gas flow with a Pitot tube, as described later.

The orifice plate and the standard nozzle, as shown in Fig. XVI–6a and 6b respectively, are very similar in their operation. The fluid is forced to take up the flow lines of a Venturi tube, but the exact area of the throat and the proper places for pressure tappings are not clearly indicated. It is necessary to choose an arbitrary position for these tappings, and in practice this is generally the so-called "corner" tapping shown in the diagram. While the standard nozzle must be made up with considerable care, the sharp-edged orifice plate shown in the diagram can often be replaced by a thin metal plate when no extreme accuracy is required. Such a plate can

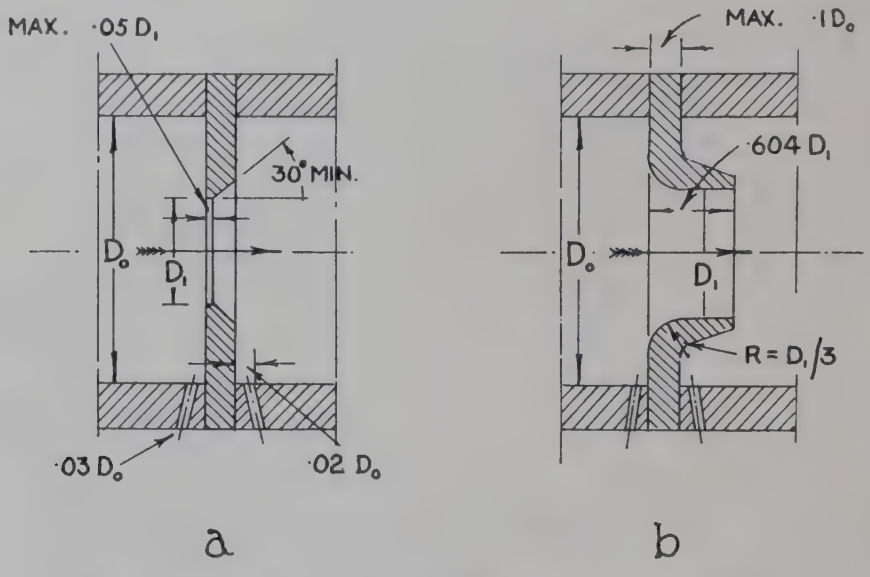


FIG. XVI–6.—STANDARD ORIFICE PLATE AND NOZZLE.

be clamped between two drilled flanges to give the corner tappings required. The formula already derived for the Venturi tube will hold in the simplified form

$$v_1 = C\sqrt{(2gH_w \cdot \rho_2/\rho_1)}$$

where v_1 multiplied by the area of the nozzle or orifice gives the volume of fluid passing, and C is an experimental correction coefficient.

The value of C varies much more widely with conditions than in the case of the Venturi tube. In the first place the coefficient for orifices varies in an irregular fashion with Reynolds Number, passing through a maximum and then decreasing to a steady value as the Reynolds value increases from 10 to 100,000. For a standard nozzle

the variation is much less marked, and values approximate to those of the Venturi. Secondly, it is possible to choose any arbitrary value for the ratio D_1/D_0, where D_1 is the orifice diameter and D_0 the conduit diameter. Although the value chosen has some effect on C with low Reynolds Numbers, there is little or no difference when the number exceeds 10,000. Standard nozzles are generally used with a ratio of D_1/D_0 which is less than 0·5, and again the coefficient follows the values for the Venturi meter. The general outline of these corrections is shown in Fig. XVI–7.

One of the chief objections to the orifice meter is that the bulk of the pressure drop is not reconverted to fluid head as in the Venturi. The loss varies with the ratio D_1/D_0, and is approximately $(H_0 - H_1)\{1 - (D_1/D_0)^2\}$. As a typical value of D_1/D_0 is 0·5, some 75% of the pressure drop recorded is lost in heat, etc. For a nozzle the loss is somewhat less, but hardly sufficient to warrant the additional expense of a standard section. The accuracy of reading is also more subject to disturbance by eddies in the upstream section than is the case with a Venturi meter, and it is desirable to arrange for some 30 diameters of straight pipe before the orifice or nozzle is introduced. This means that an ideally placed orifice meter will be nearly as long, overall, as a Venturi meter unit, but often this precaution is not followed. It is probable that the utmost accuracy that can be expected from either nozzle or orifice is $\pm 2\%$. Most improvised fluid meters in chemical plants are of the orifice type, the ease of improvisation accounting for their popularity. The great disadvantage is the irrecoverable pressure drop.

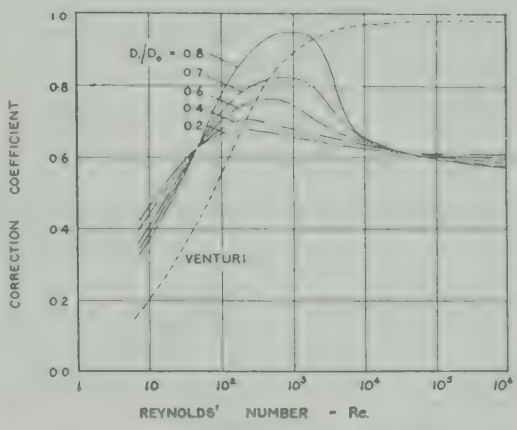


Fig. XVI–7.—Correction Coefficients for Orifice Plate.

The loss of pressure can be avoided, with some loss in accuracy, by the use of a Pitot tube. These tubes, which measure the difference between impact and static pressure, are made up in a number of ways, of which the two most common are shown in Fig. XVI–8. The impact opening (a) of the tube must face straight upstream, while the static opening holes (b) must be drilled in a smooth surface

with no roughness at the edges. The tube should be small in relation to the conduit, so as to make no difference to the essential flow lines. At any point in the fluid stream, if ΔH is the difference in pressure between the two legs of the Pitot tube, the velocity of the gas v_0 is given by

$$v_0 = \sqrt{(2g\Delta H)}$$

where v_0, g, and ΔH are in self-consistent units. Again, it is general to use a liquid such as water in the actual gauge, with a density ρ_2, while the fluid in the conduit has a density ρ_1. Then $\Delta H = H_w \cdot \rho_2/\rho_1$ and $v_0 = \sqrt{(2gH_w \cdot \rho_2/\rho_1)}$. It may be necessary to insert a correction coefficient in the formula for other forms of Pitot tube, but for those shown in Fig. XVI–8 the value will be from 0·95 to 1·05 and the accuracy of a Pitot tube is not much closer than this.

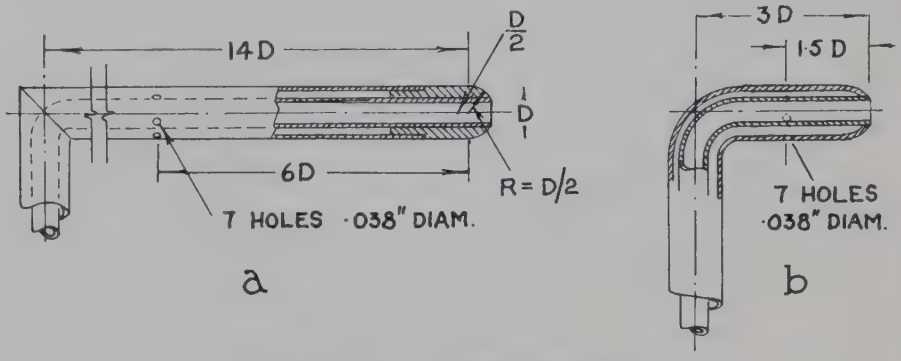


Fig. XVI–8.—Pitot Tubes.

The device measures the instantaneous velocity at one point in the conduit. As the fluid velocity across a pipe will vary, it is generally necessary to take a number of readings at different points. The cross-section of the conduit can be divided up into a number of zones of equal area. For a circular pipe these will be annular rings around a central circle. Two readings are then taken, across a diameter, in each of the sectors, and a mean velocity in each sector determined. The average velocity in the pipe is then calculated from these composite velocities. For a " ten-point traverse " of a circular pipe, with five zones, the points for readings are at 0·316, 0·548, 0·707, 0·837, and 0·948R from the centre, where R is the radius of the pipe channel. This traverse will also show whether flow conditions in the pipe are normal, a state of affairs which can usually be assured by a straight pipe length of 50 diameters before the Pitot tapping point. Any regular swirl, such as that caused by a

tangential entry, will persist for greater pipe lengths than this, and readings which show an irregular distribution of fluid velocity should be interpreted with caution. For straight pipes the relation between maximum velocity at the centre of the tube and mean velocity in the pipe varies widely with the Reynolds Number, as might be expected. A graph of the ratio $v_{av.}/v_{max.}$ against the Reynolds Number is shown in Fig. XVI-9, and again indicates the desirability of keeping flow conditions well inside the turbulent region when measurements are taken.

Measurement of velocity with a Pitot tube can be regarded as an approximate method with especial value when pipe-lines are in use. It is a relatively simple matter to open a hole in a conduit when the internal pressure is not excessive, and a compact Pitot tube can be inserted through a hole of $\frac{1}{2}$-in. size. The interference with flow is negligible, and there is no loss of static pressure, but micrometer gauges are often necessary. Thus the difference in water head for an air-flow of 5 ft./sec. is only 0·15 mm., and many air velocities of less than this have to be measured.

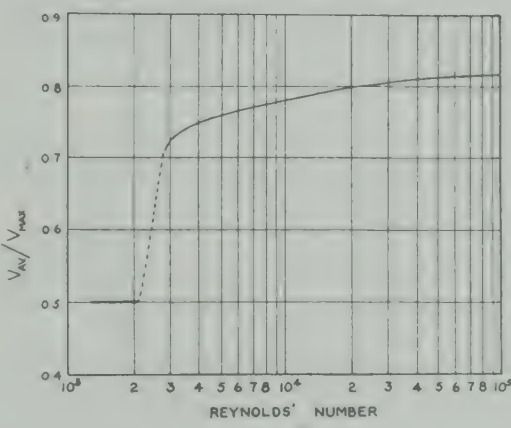


Fig. XVI–9.—Variation of Velocity with Reynolds Number.

When it is possible to obtain free access to a conduit for gases, as in the case of a stack outlet or a ventilation duct, a vane anemometer can be used instead of a Pitot tube. These are delicately made fans, connected to a counting and recording mechanism, and are calibrated down to gas speeds of as low as 0·1 ft./sec. They are fragile and soon get out of order in corrosive atmospheres, while a Pitot tube is robust and can be made up from any corrosion-resisting material. The author has used tubes in glass or stainless steel for the measurement of flow rates in nitric acid vapour ducts. The anemometer is less susceptible to slight changes in setting than the Pitot tube and is rather more accurate when it can be used.

A fan rotated by the fluid stream is the basis for a large number of measuring instruments besides the gas anemometer. A simple water meter of this type is shown in Fig. XVI–10. The paddle in

the water conduit is arranged with a long stem to keep the indicating and recording counters well out of the way of the water, and the whole of the moving parts are arranged and lubricated to give a minimum of resistance to the torque exerted by the water on the paddle. Despite these precautions there is bound to be a certain degree of " slip ", which will increase as the mechanism stiffens with drying up of lubricant, or corrosion. The use of these impulse-wheel machines will therefore call for constant careful maintenance

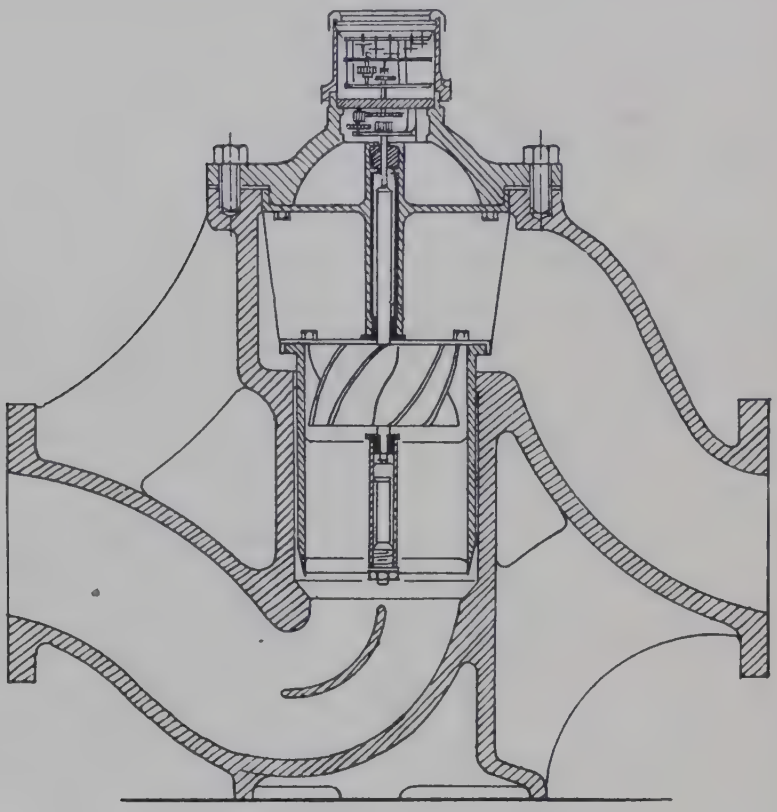


FIG. XVI–10.—WATER METER.
(Courtesy of George Kent & Co.)

if accuracy is to be maintained. When the total flow is large, it may not be desirable to pass all the fluid through the paddle chamber, and a fixed proportion is by-passed to give a more compact meter. The back-pressure thrown by such a form of measuring device is very small, and a large number of proprietary meters are available.

Positive displacement meters are not so subject to slip and are more accurate—to $\pm$ 1%—but they throw a back-pressure of some 1–2 ft. of head of the fluid passing. Such meters are reversed pumps, with the work being done by the fluid, and often take the

same shape and design as pumping units. The Holmes-Connersville meter shown in Fig. XVI-11 is identical in design with the blower marketed by the same organisation, while the water meter of Fig. XVI-12 is very similar to one of the positive rotary pumps which have been devised for pressures up to 100 lb./sq. in. These meters,

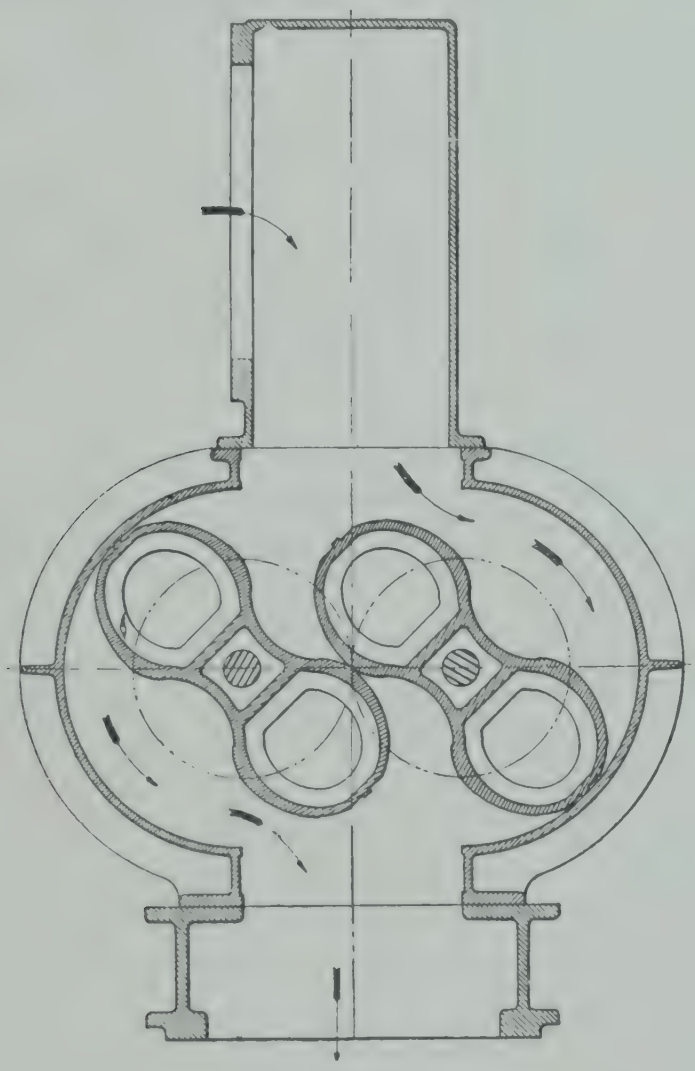


Fig. XVI–11.
(*Courtesy of W. C. Holmes & Co.*)

as well as the impulse type of the previous paragraph, rely upon a carefully constructed gear-wheel train, which is not easy to maintain in the corrosive atmosphere of the average chemical plant.

This point of corrosion-resistance is most important when very corrosive fluids, such as strong acids, have to be metered. The rotameter gauge has been found very suitable for this work. It

consists (Fig. XVI–13) of a small conical indicator moving in a tapered glass tube. As the liquid passes up through the tube, it

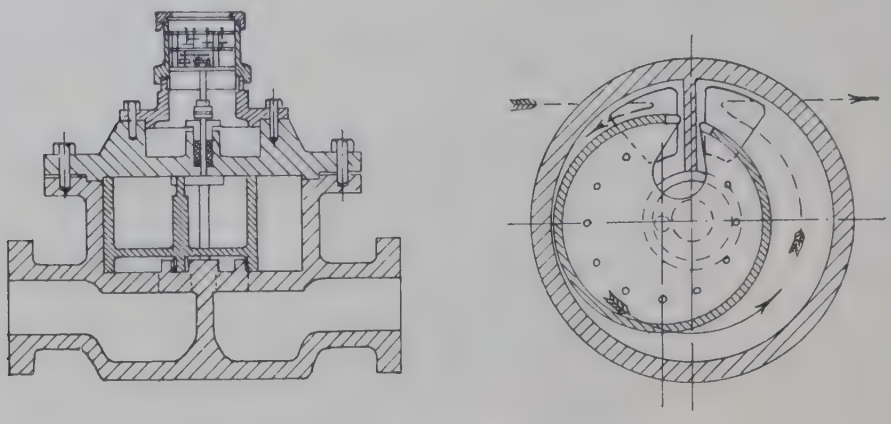


FIG. XVI–12.—WATER METER—PUMP TYPE.
(*Courtesy of George Kent & Co.*)

raises the indicator until the fluid drag, by velocity, in passing the annular channel round the indicator, balances the forces of gravity.

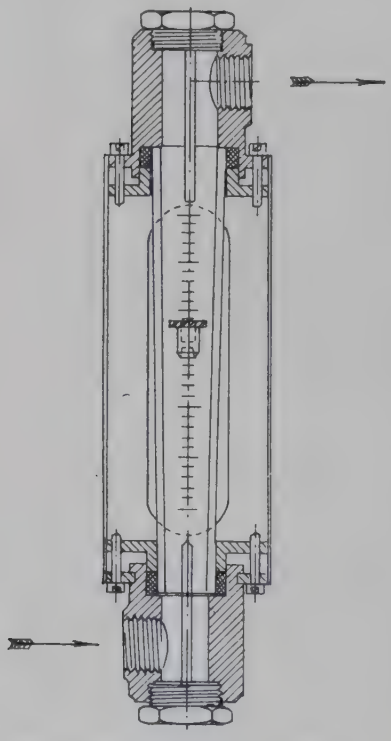


FIG. XVI–13.—ROTAMETER GAUGE.

The float and tube can be of glass, mounted in a stainless steel or synthetic resin frame, and all mineral acids with the exception of HF have been successfully metered in this way. The indicator is grooved so that the fluid flow causes a slow rotation, and this has the advantage of showing at a glance that the meter is functioning properly and that flow is occurring. The gauge also has the advantage that displacement up the tube is directly proportional to flow rates and not proportional to the square of the velocity, as in many other instruments. A rotameter can be built to give a flow range from 5 to 50 gal./hr., or some multiple of these rates, in a single 24-in. tube, and will last indefinitely provided that no deposit forms on the indicator. The real cost lies in the special tapered glass tube, which is liable to be accidentally broken. It is perhaps regrettable that the straight-sided tube,

with an inner tapered core, has not been further developed as a substitute.*

The variables measured by these instruments have all been those of quantity, either weight or volume. Other variables which must frequently be measured are those of quality—temperature, pressure, or such units as acidity or colour. The first of these is the most common controllable variable in chemical plants. For automatic control the mercury-in-glass thermometer, besides being very fragile, is not nearly so suitable as metal-sheathed systems which transmit pressure to a Bourdon tube, and thence to an indicator and/or a control element. The most popular is the vapour-pressure thermometer, in which the pressure element is filled with a liquid of suitable vapour pressure for the temperature range being recorded. The gauge responds very rapidly to temperature changes, but does not give an even scale of deflection on the Bourdon tube, owing to the irregular variation of vapour pressure. Such thermometers are especially suitable to the temperature range 0° to 350° C. Mercury-in-steel pressure thermometers can be substituted for the vapour-pressure form, and give an even deflection over the temperature range 0° 450° C., but are rather slow in response to changes. Other liquid-filled thermometers, with toluene as the most popular filling, are used from temperatures of −60° to 200°. Gas filled thermometers are unusual, but have been used between 300° and 600°: they are very slow to respond to temperature changes.

An alternative to what may be termed the mechanical devices for temperature measurement are the electrical units -the thermocouple and the resistance thermometer. The simplest of these is the former, made up usually from copper constantan (up to 300°), iron-constantan (200° 800°), chromel-alumel (300° 1100°), or platinum PtRh-alloy (up to 1500°). The e.m.f. is measured and recorded by an automatically balancing potentiometer as a rule, but for simple use any form of milli-voltmeter can be attached, with due regard to internal resistance. The resistance thermometer has to be made up from either pure nickel (−200° to 500°) or pure platinum (all temperatures up to 1700°). The resistance is measured by a potentiometer of the Wheatstone-bridge form, the operation of which can be made automatic, with the reading transferred to a recorder or control.

Absolute-pressure instruments are almost invariably of the

* Stout and Rowe, *Trans. Amer. Inst. Chem. Eng.*, 1938, 34, 1.

Bourdon-tube form, which is easily attached to a recorder or to a control device. For low pressures a diaphragm similar to those used in barometers can be used, though the very small deflections need an accurate magnifying mechanism. When the total pressure is less than 1 mm. of mercury, it is necessary to use special vacuum gauges of the Pirani or ionisation type. These instruments, as with those used to measure acidity or colour, are so highly specialised that recourse should be made to specific publications on the subject. The measurement of liquid levels in tanks is very conveniently made by measuring the pressure required to blow air from an orifice at the tank bottom. If two orifices are used, at a fixed distance apart, then the pressure difference is a measure of the fluid density, and may be used to control rates of flow of constituents to a mixing chamber.

CHAPTER XVII

AUTOMATIC CONTROL

While it is obvious that automatic control is not confined to the chemical industry the use of instruments to control the operation of chemical plant is increasing steadily, and as the demands on labour grow, the increase may be still more marked. It has already been urged that any new chemical plant shall be built around an efficient control system, with primary variables properly chosen and the plant design modified accordingly.* The cost of such a control may be from 1% of the total plant cost, when making heavy chemicals, up to 15% in the case of complicated organic syntheses.† The total cost of remote control on plant making radioactive material is unknown, but must be even higher than this last figure. The instruments must be overhauled weekly by a competent technician but still cost, on the average, less than a tenth of the labour charge that is saved.‡

Any control system consists of a measuring instrument and a control activated by signals received from that instrument. No measuring device can give an instantaneous response to changes in the condition which it is measuring. As an example, suppose a thermometer in a tank of water, indicating a temperature θ_0. If a quantity of hot water is added with efficient mixing, the temperature of the tank changes very rapidly to θ_1. The thermometer indication changes more slowly, giving the value of θ_1, at the end of an indefinite time period. The change $D\theta$ at the end of a time t will be given by Newton's Law as

$$D\theta = (\theta_1 - \theta_0)(1 - e^{-t/L})$$

where L is a characteristic of the measuring instrument. When $L = t$, numerically

$$D\theta = (\theta_1 - \theta_0)(1 - e^{-1}) = 0{\cdot}632(\theta_1 - \theta_0)$$

* See, e.g., Van Antwerpen, *Ind. Eng. Chem.*, 1942, 34, 387.
† Considine, *Chem. Eng.*, 1947, 54 (No. 7), 108.
‡ Comstock and Fisher, *Trans. Amer. Inst. Chem. Eng.*, 1947, 43, 303.

The value of L, defined as the Lag Coefficient, is thus given by the time required for the measuring instrument to record 63·2% of the total change. Typical lag coefficients are as follows:

Pressure Thermometer in Liquid	0·10 min.
Pressure Thermometer in "Well"	1·66 „
Thermocouple in Gas Stream	0·58 „
Mercury Manometer on Flow Meter	0·04 „
Resistance Thermometer—Potentiometer	0·03 „

It has so far been assumed that conditions remain constant after an initial change. Normally conditions will go on changing, and the recorded value will lag steadily behind the actual figure. For a steadily rising temperature, such that $\theta = \theta_0 + kt$, it can be shown that the recorded temperature $\theta_0 + D\theta$ will always be lower than

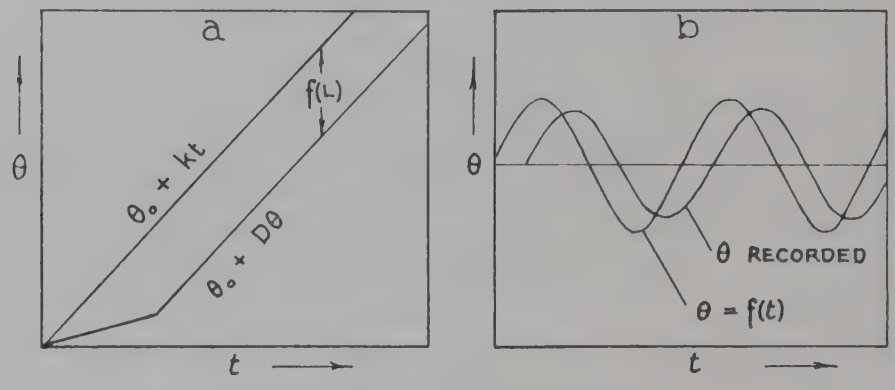


Fig. XVII–1.—Instrument Lag.

the true value by a dynamic error $f(L)$, which is steady after an initial disturbance has died away, as shown in Fig. XVII–1a. Where the temperature varies in a cyclic fashion, so that $\theta = f(t)$, as in Fig. XVII–1b, then the measured value will form a curve of the same period but of lower amplitude. lagging behind the actual value. All control is subject to these limitations of the measuring instrument, which are no less marked for other variables than for temperature. A further shortcoming is that the instrument can only be fixed at one place. Thus the pressure gauge on a boiler registers the pressure at the inlet of the gauge connection, while the temperature of a fractionating column is measured on one plate. This will form a serious defect if the relation between the measured "zone" and the rest of the plant changes, and is in any case a contributor to the "process" lag of a control system. This process lag may be simply illustrated by considering the case of the water tank to which

hot water is added without efficient stirring. Then the thermal disturbance may take some time in reaching the thermometer bulb, after which the ordinary delays in recording will still occur. It must also be noted that for all measuring instruments, with the possible exception of some electronic devices, there is a small " dead zone " through which the variable may change without the alteration being recorded by the instrument.

The signals of the instrument are transmitted in a number of ways to a controller, which operates to check and adjust changes in the variable. In the form which is familiar to all technologists, an electric heater is turned on and off by the current from an electrical circuit made or broken by a mercury thread moving in a thermometer. This ON-OFF method, as represented in Fig. XVII–2a, is

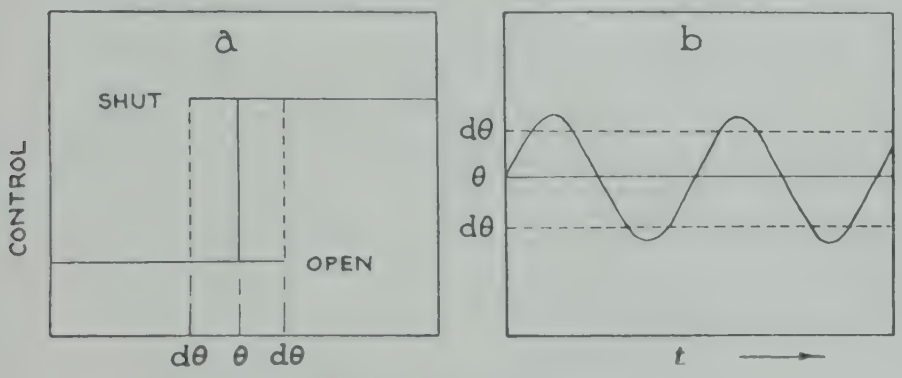


FIG. XVII–2.—ON-OFF CONTROL.

the simplest of all forms of control. At all values of the variable below the arbitrary value θ the control is full open—resulting, in the example above, in full heating current passing. At all values above θ the control is full shut and no heating current passes. The simple continuous line of Fig. XVII–2a represents an ideal control which can never be attained in practice, as the " dead zone " on the measuring instrument will close the control at $\theta + d\theta$ and open it at $\theta - d\theta$, as recorded. These recorded values will still be lagging behind the actual values, as indicated above. It can be assumed that any change of temperature will be well within the capacity of the heater, and the changes of control indicated by the dotted lines of Fig. XVII–2a will result in a variable fluctuation as shown in Fig. XVII–2b. The variable will move outside the limits $\theta - d\theta$ and $\theta + d\theta$ to an extent which is determined by the lag of both recording instrument and of controller, though this last, for electrical devices,

is generally small. The period of the oscillations shown in Fig. XVII–2b will be determined by the relative capacity of the controlling devices and the process which is being controlled. For example, a tank may be filled with liquid to a steady level by a constant-speed pump, actuated through a relay by contacts in a mercury-level recorder. The joint lag of this recorder and control will allow an arbitrary oscillation of 6 in. up or down from the desired level. If the capacity of the pump is such that the control level is restored in 1 min., while the " process demand " drops the level 6 in. in 5 min., then the level will oscillate in the way shown in Fig. XVII–3a. If the capacity of the tank is increased, the period of the curve will increase, while increases in the rate of filling and emptying will decrease the period. They will also increase the amplitude, as a lag is always in *time*, which translates into different

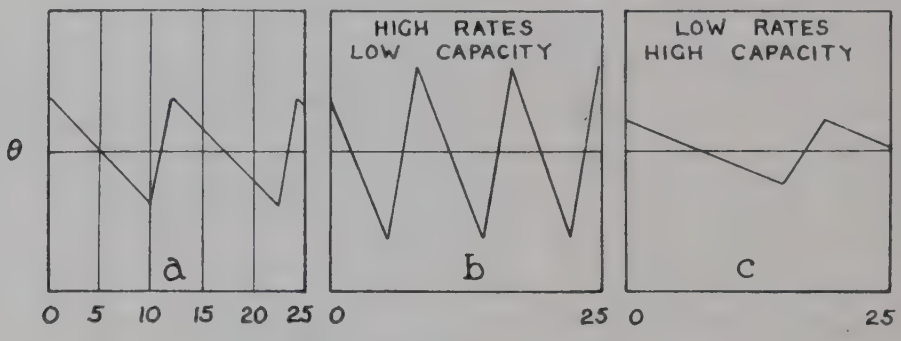


Fig. XVII–3.—Variation in On-Off Control.

level variations according to liquid rates. The effects of these changes are suggested in the diagrams of Fig. XVII–3b and 3c, and it will be seen that in all cases the effect on ON–OFF control is to produce a cyclic movement of the variable about an arbitrary point or setting.

If alterations in the variable produce a steady change in the controlling device, the process is defined as being under *floating control*. For example, a tank of water may be fed from a pressure line closed by a motor-operated valve, which is set in motion when high or low control levels are reached, and cuts out, holding the valve stationary, when the " dead zone " around the control point is attained. The operation of this system is shown in Fig. XVII–4. The rapid changes of simple ON–OFF control are avoided, as the control is partly adjusted to the load, but oscillation between high and low levels must still go on, and a sudden change in demand can

give a surge in level well outside the normal limits of the " control band ". The general form of such an oscillation is shown in Fig. XVII–4b.

Such surges may be largely overcome by varying the speed of the valve movement according to the position of the variable, a method defined as *proportional speed floating control*. The motor moving the valve will now start to work whenever the variable moves outside the neutral range in either direction, and will reach full speed as the variable reaches some arbitrary position. The " floating rate " of such a control is defined as the percentage of valve motion per minute for unit deviation of the variable. Thus, for a tank of water being heated by steam through a controlled valve, a " floating rate " of 50% per minute for a 1° deviation would mean that the valve

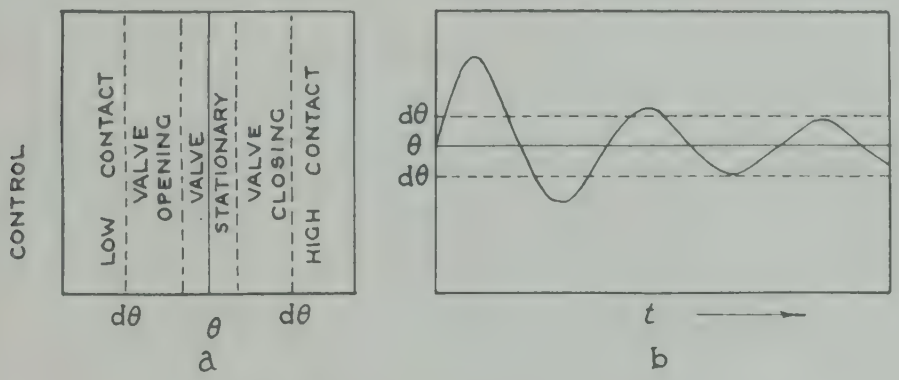


Fig. XVII–4.—Floating Control.

closes completely in 30 seconds if the tank temperature reaches a value of $\theta + 4$ C. The temperature will then fall until the low side of the neutral band is reached, when the steam valve starts to reopen, with speed of movement increasing steadily as the deviation increases. It must be borne in mind that in all cases of control there must be sufficient energy available to cope with the largest load put on the process (i.e. in the example above, the temperature of the tank must rise when the steam is full on). The form of the temperature–time curve of the tank under proportional-speed floating control will be as shown in Fig. XVII-5. This curve is very similar in form to ON–OFF control data, but the amplitude is more closely controlled, while high capacity remains in reserve in case of a sudden load surge. Cyclic variations around a mean position remain, and the valve setting tends towards full open or full shut whenever the motor is set in motion.

In order to overcome this cyclic movement of the variable, the control may be arranged to move from a full open to a full shut position as the variable changes through a " proportional band " of its full possible change. In a water tank, with a controlled liquid depth of 5 ft., a proportional band of 20% would mean that the valve

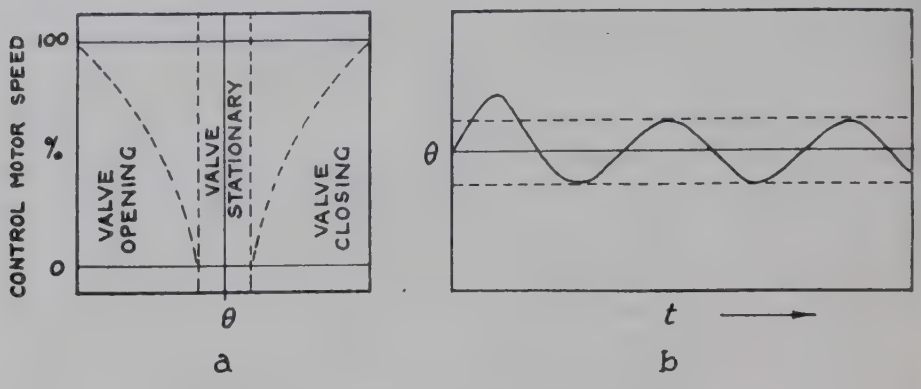


Fig. XVII–5.—Proportional-Speed Floating Control.

controlling supply would move from full open to full shut as the level rose from 4 ft. to 5 ft. Similarly, a 100% proportional band on water being heated by steam in an open tank would mean that the steam valve is full open at 0° and full shut at 100° C. The movement of the control in this fashion is defined as *proportional response*, and may be represented by the diagram of Fig. XVII–6a. The valve

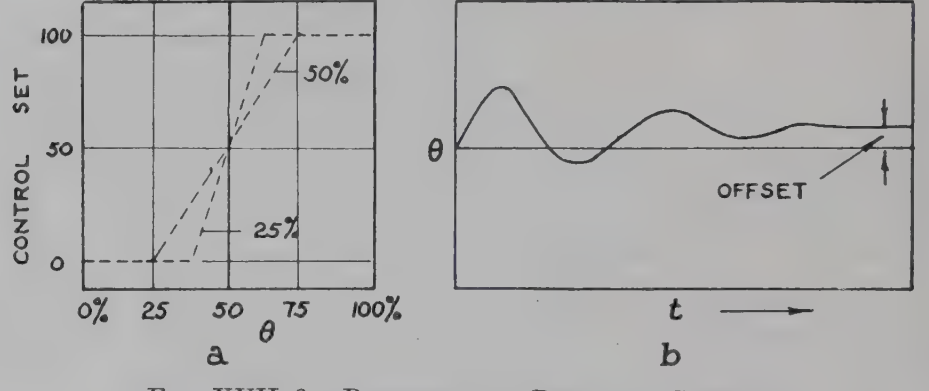


Fig. XVII–6.—Proportional-Response Control.

is shown in the half-open position as the variable passes the control point. This is not necessary, and the control may be arranged as full open or full shut at this point, according to process requirements. The rate of control movement as compared to the rate of change of the variable is defined as the *response* of the control. For a 25% proportional band the response will be 4 to 1, and so on.

The effect of proportional-response control is to increase the opening or closing of the control device until the variable is held at a steady value, but this value will always tend to be above or below the setting point. Consider the case of a water tank, with the liquid level determined by a control valve set at 50% open for the ideal level θ, and with a proportional band of 20% of the tank depth. Then an increase in outlet flow or load will cause the control to open the valve to some point—assumed at 60% open—which gives a balance between inlet and outlet flow. The level in the tank, however, will have dropped to the point which corresponds to a 60%

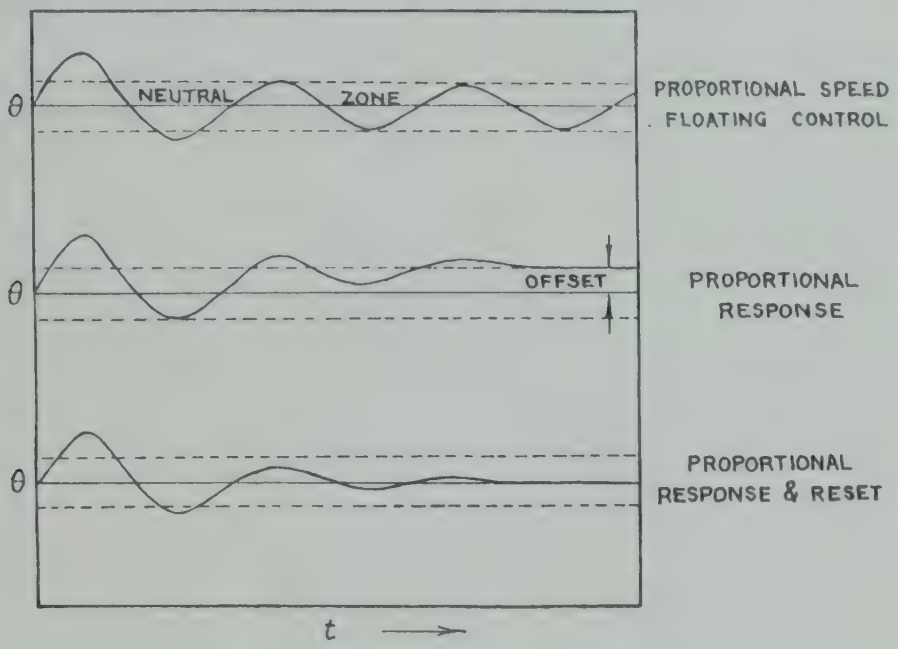


FIG. XVII–7.—CONTROL COMBINATION.

open valve, or for a 20% band a drop of 2% in the total depth. The change in the variable is shown in Fig. XVII–6b. This drift from the set, known as *offset*, will be increased by a wide proportional band, or low response, but if the proportional band is made too narrow, lag phenomena will produce cycling as with an ON–OFF method.

If a variable is governed by proportional response it will tend to take up a steady value, away from the ideal setting point. Floating control and ON–OFF methods give a cyclic variation around this setting point. A combination of the two, known as *proportional response with reset*, will give the ideal control method in which the variable is brought back to a steady state at the setting point. The three are represented in the diagram of Fig. XVII 7, showing

the method of proportional-speed floating control combined with proportional response. The two control systems must be so arranged that the offset of the latter is outside the neutral zone of the floating control and the consequent complexity of the control mechanism is considerable.

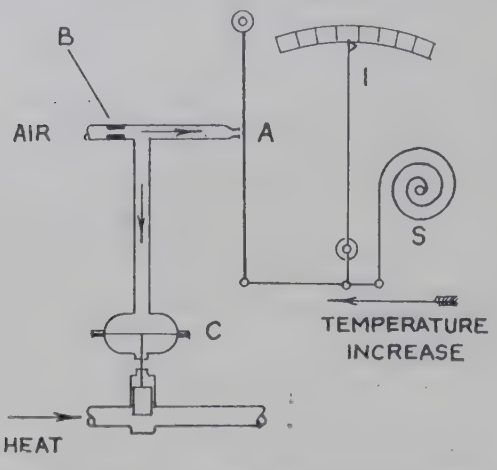


FIG. XVII–8.—TEMPERATURE CONTROL BY AIR-OPERATED VALVE.

A wide variety of automatic controllers applying the above principles with all forms of chemical plant are now available. Many are highly specialised, but they fall generally into two main classes, those of pneumatic and those of electrical operation.

A pneumatic-control instrument is shown diagrammatically in Fig. XVII–8. The temperature-measuring device is a pressure thermometer connected to the Bourdon tube S, which in its alterations of shape with temperature opens or closes an air outlet valve at A. The air supply to this valve passes through a small orifice at B, in amounts which can easily escape at A, until the "flapper" activated by the Bourdon tube closes the outlet. When this occurs, the pressure in the air pipe increases, and starts to close the pressure-operated valve C. If the mode of operation of the "flapper" is very sharply defined, so that the valve is either full open or full shut, then the arrangement of the figure will approximate to ON–OFF control. It is not easy to make the movement of the valve so definite, and the general tendency is for the air pressure on the valve C to build up to an intermediate value, holding the valve so that input and output of heat are balanced—not necessarily at the setting point. This principle is accepted and accentuated by a "feed-back" bellows as shown in Fig. XVII–9; the air pressure building up behind the closed flap valve moves the bellows head to force the flapper away from the air outlet. This combined action tends

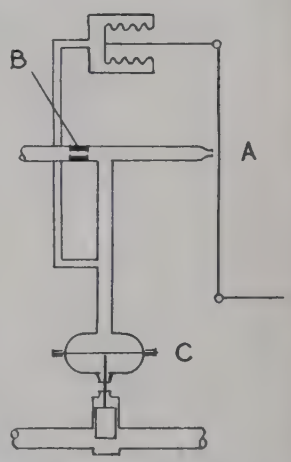


FIG. XVII–9. — PNEUMATIC CONTROL WITH "FEED-BACK" MODIFICATION.

towards a proportional response control. It is possible to reverse the valve action, and arrange for increase of temperature to open the flap valve, allowing air to escape, when the valve C opens under the pull of a spring instead of closing. In any control system it is desirable that the variable shall be recorded, and this is easily arranged by attaching a link mechanism to the Bourdon tube and transferring the movement to a pen and chart, as at I in Fig. XVII–8. A second bellows, with an extra air chamber attached, can be arranged to act in the opposite direction to the first "proportional-response" bellows. A small air leak from the controlled-pressure air line into these "reset" bellows will cause a movement of the

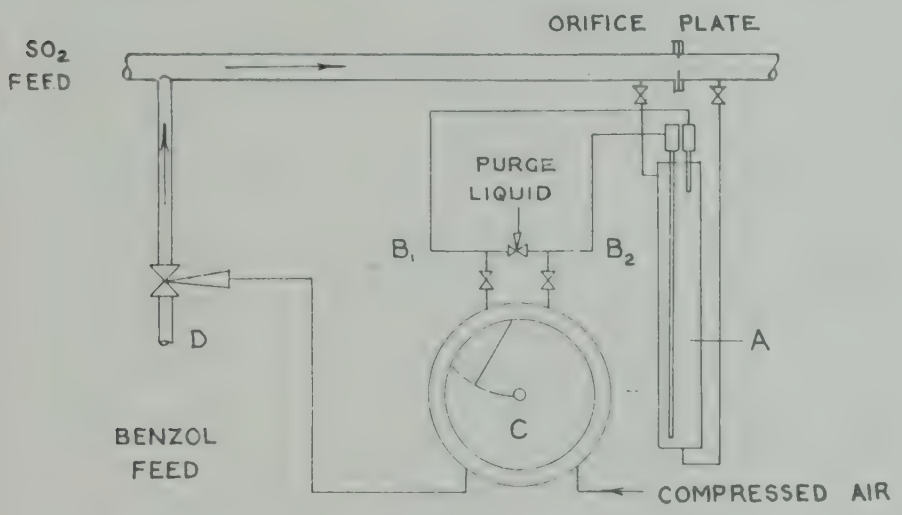


Fig. XVII–10.—Flow-Rate Control—Air Operation.

flapper in the same direction as the last movement caused by the controlled variable. Thus, if this has caused an increase in the pressure of the air, then the reset gives a further increase. Due adjustment of the speed of working of this reset can be made by altering the magnitude of the air leak, and a complete control of the variable can be achieved.

Automatic control of flow rates can be achieved in the same way. In Fig. XVII–10 an orifice constriction on a pipe-line forces a small bye-pass flow of mixed solvent (benzol and sulphur dioxide) through a measuring chamber A.* The density of this mixture determines the differential pressure across the lines B_1, B_2 supplying small quantities of light oil to the process. This differential pressure is

* Pull, *Trans. Inst. Chem. Eng.*, 1945, 23, 158.

recorded on the dial C and actuates an air-release valve to set a benzol feed rate through the side feed line D to give a regular density to the mixed solvents. The small quantity of light oil which is used to record pressures has the added advantage that the corrosive and extremely volatile SO_2 is kept from any delicate recording mechanism. This continuous purging of pipe-lines is widely used in all controlling and recording processes of the non-electrical type to ensure that connecting lines do not block or corrode. For instance, it is common practice, when measuring pressures in large vacuum stills, to arrange for a small air leak at the instrument panel. The air passes continually into the plant, keeping conden-

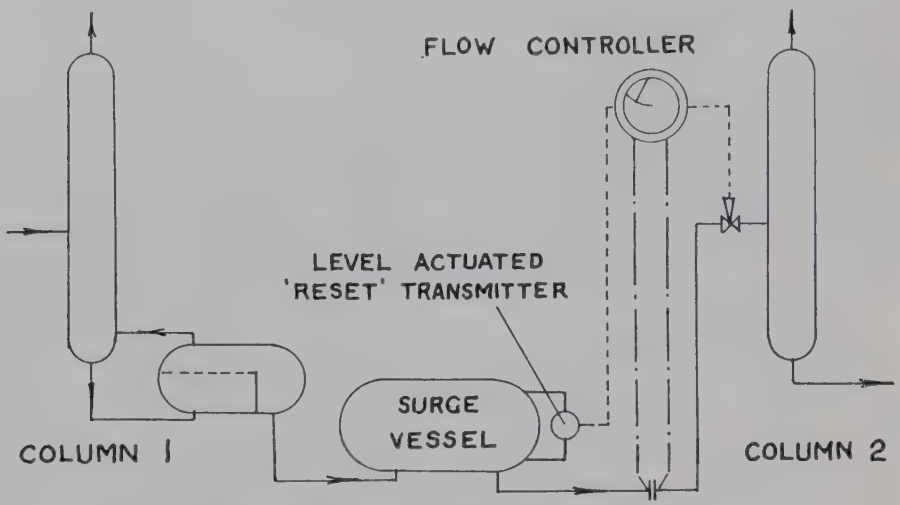


FIG. XVII–11.—"RESET" CONTROL.

sates out of the long leads, and giving, by small bubble indicators, a visual assurance that the leads are clear.

Despite these precautions, the long pipe-lines associated with pneumatic-control methods are a manifest disadvantage. Ice blockage is often met with in cold weather unless the compressed air is dried and free from mists, and long thin tubes are always liable to be accidentally blocked. Nevertheless, a recent review of control systems showed that pneumatic control was more popular than the use of electrical impulse units. This was perhaps owing to the ease with which diaphragm-operated valves can be moved by compressed air. Special forms of valve are used for these controls, with throughputs which are as nearly as possible proportional to the amount of valve movement.

The use of a double control to give reset is represented in Fig. XVII–11. This shows the working of two fractionating columns

in series, with residues from the first passing through a surge vessel to an orifice plate activating a flow control. A differential pressure level measuring device is connected to a control mechanism which alters the control band or setting point of the flow controller. Then a surge of liquid from the first column will be held up by the flow control until the raised level in the surge tank has adjusted the setting point to a suitable figure.

Despite the present popularity of pneumatic operation, it is probably that electrical operation, with the advantage of reduced lag in the control and the growing knowledge of electronic devices, will ultimately replace all other methods. A simple control circuit

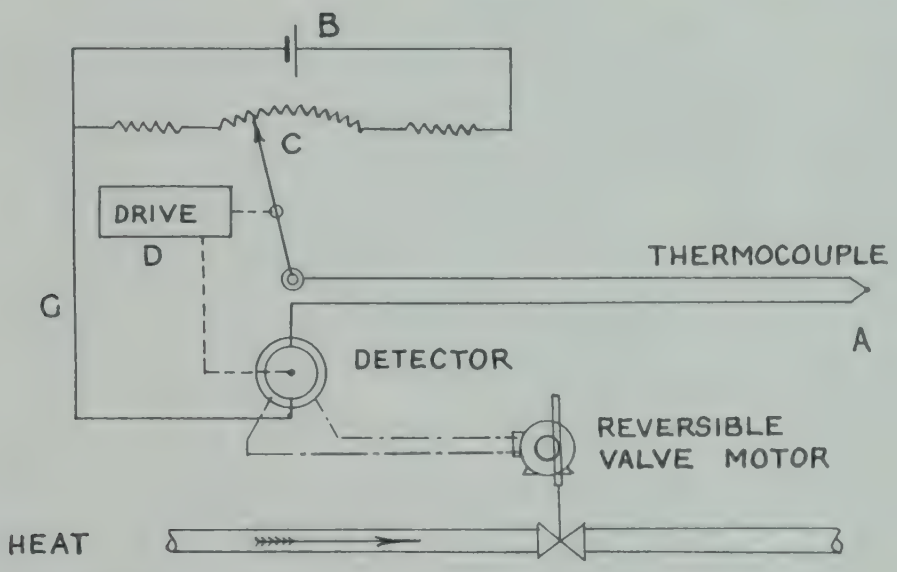


Fig. XVII-12.—Temperature Control—Electrical Operation.

based on the e.m.f. from a thermocouple, is shown in diagram at Fig. XVII-12. The potential of the thermocouple is balanced against a cell B at some point on the slide wire C, so that no current flows in the control circuit G when the thermocouple is at a predetermined temperature. If this temperature changes, the circuits on the slide wire are no longer balanced, and a current flows through the circuit G. This is picked up by a valve detector and amplifier, which sets in motion a reversible motor D and moves the thermocouple connection over the slide wire to bring battery and thermocouple back into balance. At the same time another motor circuit has been activated by the amplifier and has moved a valve to increase or decrease the supply of heat to the process.

Considering the condition in which the motorised valve is half open for normal operation and temperature at the control point, a slight increase in load will give a fall in temperature in the process, followed in due course by an out-of-balance e.m.f. The motors are set in motion to move the indicator to a new position of balance and to open the motorised valve to some extent. If movement of the valve is slow compared to that of the indicator, the process settles down to a new balance " offset " to the first, a typical example of proportional response. On the other hand, the motorised valve may be very fast, tending towards full open or full shut directly the control diverges from the set point. As full heating capacity is more than equal to the largest possible load, this will give an ON–OFF control with cycling around the control point. The amplitude and period of the cycling will be determined by the relative capacities of the process and the heating system and by the lag of the controls. As a further development, the motor circuit may be driven by a proportional-speed floating control, thus checking any wide surges of temperature under a suddenly changing load. The relation between these movements is too complicated to be dealt with here, and recourse should be made to specific treatises on the subject.*

All automatic control methods need careful and regular maintenance if they are to be of service. A sticking pen on a chart is a frequent cause of temporary disturbance, while a diaphragm-operated valve should be kept as clean as possible for the same reason. It seems that the ultimate limiting factor in any control is the lag of the measuring instrument, even when pneumatic operation of the control mechanism is used. In such operations as milk pasteurisation the lag should not be more than 5 sec., and with such rigid conditions the position of the control valves relative to the rest of the plant must be carefully considered.† Although the flow of current to an electric motor is almost instantaneous, there must always be a slight mechanical inertia to slow up the action of a motor-operated valve. The balancing of such factors as these will need to be specific to the process being studied.

The chemical industry is always up against health problems, because so many of the operations are carried out under unhealthy conditions. Automatic control will be essential in these operations in the future, because there will be no labour available and willing

* See, e.g., Eckman, " Industrial Process Control ".
† Hill, *Trans. Inst. Chem. Eng.*, 1945, 23, 165.

to work in them. The growth of processes in which radioactive chemicals are being handled is likely to be phenomenonal in the next few years, and it is probable that all branches of automatic control will develop equally rapidly. For these reasons the final section of this book may well refer to the most important of all the mechanical aspects of chemical plant operation.

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